CHARACTERIZATION OF DUCTILE IRON THROUGH FRACTOGRAPHIC STUDY

Thesis submitted in partial fulfilment of the requirements for the award of the degree of

Master of Technology

in

Mechanical Engineering

Submitted by

BISHNU PRASAD MAHTO

Roll No.-**212MM2456**

[Specialization**:** Steel Technology]

Department of Metallurgical and Materials Engineering

National Institute of Technology

Rourkela-769008

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Under the supervision
of
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CERTIFICATE

This is to certify that the work in this Thesis Report entitled "**CHARACTERIZATION OF DUCTILE IRON THROUGH FRACTOGRAPHIC STUDY**" by **Bishnu Prasad Mahto** has been carried out under my supervision in partial fulfilment of the requirements for the degree of Master of Technology in Steel technology during the session 2012-2014 in the department of Mechanical Engineering, National Institute Of Technology, Rourkela.

I appreciate his presentation during the project period. He completed the project successfully as per the requirements.

I wish him success in all future endeavours.

Place: Rourkela

Date:

Prof.Sudipta.Sen

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ABSTRACT

Ductile iron is a special type of cast iron family which differs from other cast iron in the manner of ductility since others are brittle in nature. Ductile iron (DI) is gaining its popularity in many industrial applications due to its strength and considerable amount of ductility which is because of the presence of spheroidal graphite in microstructure. Fracture is very common in almost every industry and field of application and analysis of fracture now-a-days has become essential for optimizing the product life span. The current study is focused no investigating the mechanical properties and fracture characteristics of ductile iron subjected to various heat treatment processes. Tensile and impact specimens are machined from a test block according to ASTM E8 and ASTM D256 standards respectively. Specimens are austenitized at 1000°C, followed by different rate of cooling and quenching. The austenitizing time being 90minutes and quenching media are mineral oil, air and salt bath for tempering, normalizing and austempering processes respectively. Isothermal annealing is also carried out in some specimens to have comparison between mechanical properties and behaviour of the material. The tempering and austempering temperature is 500°C and time being 2hrs and 4hrs respectively. Tensile test has been performed using INSTRON-1195 and Izod Impact test is performed using Izod impact tester. Vickers's hardness is determined by application of 20 kg with 10sec. dwell time using Vickers's Hardness Tester. Fracture surfaces of each heat treated and as-cast specimens, after tension and impact test are observed under Scanning Electron Microscope. Tensile strength is found to be maximum for tempered and hardened specimen whereas annealed specimen is having more ductility at the expense of strength. The annealed specimen is found to be ductile in nature whereas the tempered and hardened and normalized specimens have showed mixed mode of failure.

Keywords:-As-cast ductile iron, Heat treated ductile iron, Microstructure, Austenizing temperature, Nodularity, Graphite Area Fraction

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Introduction

1.1. Introduction

Spheroidal graphite iron is also known as nodular iron or ductile iron. This is obtained by making small ladle additions of a modifier such as magnesium or cerium to liquid metal. Unlike flaky graphite in grey cast iron, Spheroidal graphite does not weaken the matrix considerably. For this reason, the mechanical properties of SG iron are superior to grey iron. [1] Ductile iron has very good damping characteristics and impact strength. Because of its mechanical characteristics ductile iron is widely used in various industries' equipment. Required characteristics can be impart in ductile iron using various heat treatments i.e. austempering, tempering annealing and normalizing. Various Heat treatments are carried out to impart required matrix/phase in the specimen. Different matrix has different mechanical properties. Presence of phases is confirmed by microstructures observed in metallurgical optical microscope. Austempered and hardened ductile iron has better required mechanical properties than conventional ductile iron. Due to the vast area of application, characterisation of ADI got importance. Fractographic analysis is one of the methods of characterisation of material.

Annealing treatment increases the ductility at the expense of strength and hardness by transforming the parent matrix into fully ferritic, whereas high strength and hardness can be obtained by quenching the specimen into a salt bath (austempering) from the austenitizing temperature resulting in formation of upper or lower bainitic structure depending on the cooling rate. Mechanical properties of ductile cast iron, like UTS and hardness increases with the increase in pearlite content and on the other hand ferritic matrix leads to increase in ductility and impact strength [2,3].

1.2.HISTORY OF DUCTILE IRON

It was Chinese who first invented cast iron in the 5th century BC by archaeologists. To make ploughshare, pots and weapons they poured the cast iron into the mould which is the replica of the desired component. Because of its cheap rate and easily availability it was in wide use in ancient China. However, its strength was inferior than steel. Cast has high compressive strength and it is brittle in character. Because of brittle character it less used in purposes where a sharp edge is required. Cast iron is strong under compression whereas weak under tension. Castings were not available till 15th century in western countries. Henry VIII initiated the casting of canon in England. Before iron cannons, English were

using bronze cannons. After the advent of casting technique, cannons being started to be made of cast iron. However, cannon made of cast iron is heavier than bronze. Thomas Newcomen developed the steam engine which gave a huge market of cast iron which was considerably cheaper than brass which was earlier used raw material for making engine cylinders. It was Keith Millis who invented ductile iron in 1943 which is also known as nodular cast iron/spheroidal graphite iron/SG iron. Ductile iron has more impact and fatigue resistance, because of nodular graphite inclusion, as compare to other varieties of cast iron. It was on October 25, 1949, Keith Dwight Millis, Albert Paul Gangnebin and Norman Boden Pilling got US patent for ductile iron production via magnesium treatment. [4]

In Victorian time's items like street lamps, railings, window frames etc. were made of cast iron. Now it is exclusively used for engineering purposes. Development in the field of cast iron is dynamic in nature i.e. it has developed to highly sophisticated levels. SG cast iron, compacted graphite irons are the examples of developed form of cast iron. [5]

Crude pig iron along with the remained scraps is melted and necessary adjustment in composition, as per the demand, is also done in this melting stage. For melting the raw materials nowadays high frequency induction furnace are frequently used to give chemical cleanliness, lessen sulphur contamination by not coming with direct contact to burning coke unlike in cupola melting. For this reason electric melting process is taking momentum. [5]

Because production costs of pig iron are relatively low as compared with other alloys, and since no expensive refining process is necessary, cast iron is a cheap metallurgical material which is particularly useful where a casting requiring rigidity, resistance to wear or high compressive strength is necessary. Other useful properties of cast iron include:

1. Good machinability when a suitable composition is selected
2. High fluidity and the ability to make good casting impressions
3. Fairly low melting range(1130-1250°C) as compared with steel
4. The availability of high strengths when additional treatment is given to suitable irons, e.g. spheroidal-graphite iron, compacted-graphite irons or pearlitic malleable irons.

The structure and physical properties of a cast iron depend upon both chemical composition and the rate at which it solidifies following casting.[5]

1.3. VARIOUS GRADES OF SG IRON ACCEPTED AS PER INTERANATION NORMS

The present ISO standard of ductile iron, ISO 1083-2004, classified on the basis of one of the mechanical properties such as tensile strength, hardness elongation or Brinell hardness. Tensile strength is the common basis for the Material Designation or characterisation and shown in the table 1.1

TABLE 1.1: Designation of Ductile Iron

Material Designation	Tensile Strength N/mm²	Elongation %
ISO 1083/JS/350-22/S	350	22
ISO 1083/JS/400-18/S	400	18
ISO 1083/JS/400-15/S	400	15
ISO 1083/JS/450-10/S	450	10
ISO 1083/JS/500-7/S	500	7
ISO 1083/JS/550-5/S	550	5
ISO 1083/JS/600-3/S	600	3
ISO 1083/JS/700-2/S	700	2
ISO 1083/JS/800-2/S	800	2
ISO 1083/JS/900-2/S	900	2

1.4. CHEMICAL COMPOSITION

1.4.1. Effect of composition

The effect of composition may be sub-divided as shown below:

1. Influence of the normal base iron composition.
2. Desired residual magnesium content.
3. Influence of trace elements.
4. Influence of alloying elements.

1.4.2. Influence of normal base iron composition

(a) **Carbon**: Carbon percentage normally varies between 3.0 to 4.0 percent. Though the most practiced carbon percentage varies between 3.4 and 3.8 percent. Castability, which is improved by improving fluidity, is the one of main parameters which gets influenced by a change in carbon percentage. Interdendritic shrinkage is caused during the final stage of solidification. The interdendritic shrinkage can be sort out up to great extent by increasing carbon percentage. To arrest this shrinkage defect, an equation has been derived which is followed to insure minimum shrinkage, as follows:

$$\boxed{\text{Total Carbon}+\frac{1}{7}\,\text{Silicon}=3.9}$$

Mechanical properties are less influenced by the varying carbon percentage within the desired range of SG Iron. UTS of SG Iron reduces by about 2.48 MPa per 0.1% increase of carbon while significant reduction in yield strength. Hardness reduces by about 5 numbers per 0.15% addition of carbon whereas percentage elongation or ductility increases more particularly in case of as-cast specimen. Modulus of elasticity of specimen is affected by the carbon percentage in proportion to volume of carbon in the matrix.

(b) **Silicon:** It is used as graphitizer and it increases the spheroids present in the matrix of the SG Iron. It increases the ferrite area fraction by reducing primary carbides and pearlite. Mechanical properties of SG Iron are greatly influenced by the silicon content. For each 0.25% silicon addition, at 0.18% manganese, there is increase of 21 MPa in UTS of SG Iron by strengthening ferrite matrix while reducing percentage elongation in annealed ferritized microstructure. Silicon content and temperature, controls the impact resistance. Increase in silicon or decrease temperature will reduce the impact value. 2.25% silicon content gives full ductility in the temperature -10^0C whereas when it is controlled to 1.4%, the material can be ductile to even as low as -60^0C. More general, silicon content is maximum 2.4% whereas less than 2.25% when impact has given more importance. Silicon above the normal range promotes excellent heat resistant qualities. Silicon acts as a inoculants in the production of SG Iron. To ensure minimum carbides in as-cast specimen, 0.6 to 0.8% silicon, as a result of inoculation with ferro-silicon, plays as very important role.

(c) **Manganese:** It acts as an alloy, refines pearlite and stabilizes, thus it decreases ferrite in as-cast specimen. It stabilises carbide and prevent the breakdown of pearlite to ferrite and prolongs the annealing cycle for production of ferritic structures. The desired maximum, therefore, this element is 0.5%, although slightly higher percentage will be permissible in the fully pearlitic grades. Tensile and yield strengths increase with increase in manganese content in as-cast specimen, particularly in annealed specimen. In normalised specimen, manganese provides good amount of hardenability. Manganese at 1.0% with 1.0% Nickel allows a 7 inch thick section to be normalised to 300 B.H.N.

(d) **Sulphur:** Removal of sulphur, below 0.018%, is very important and essential part of the production of SG Iron. Sulphur reacts with magnesium and gives magnesium sulphide which entraps in the casting, drosses and impairs the casting quality. Its content also affects the spheroidization of graphite which is promoted by residual magnesium whose quantity is affected by sulphur. Therefore, desulphurisation is very much essential to prevent the dross inclusion.

(e) **Phosphorus:** As in grey iron, phosphorus forms the brittle phosphide (steadite) network. It affects ductility and toughness very badly and reduces the impact resistance. Phosphorus, particularly above 0.08%, raises the brittle tough transition temperature range. Phosphorus is therefore kept lower than 0.043% in most of the castings.

1.4.3. Residual Magnesium

Magnesium combines first with the excess sulphur in the base iron to form Mg_2S till it is removed to below 0.015%. The magnesium sulphide formed readily floats to the surface where it can and should be removed. However, excess quantities of magnesium sulphide slag resulting from higher sulphur contents in the base iron may find their way into the castings giving rise to the dross defect. Further it has been found that high sulphur content in the base iron entails higher minimum residual magnesium contents required for satisfactory spheroidal graphite formation. Because of the fading effect of magnesium with holding time of the treated molten metal, depending upon the expected time of liquid metal disposal, suitable minimum residual magnesium contents have to be aimed at for treatment. If this leads to correspondingly high residual magnesium contents especially in the initial castings sure, it can lead to inverse or centreline chill. Thus judging from all angles, effective desulphurisation to as low a level as is practicable is a must in the production of S.G. Iron. When the base iron is effectively desulphurised to below 0.01%, Residual magnesium contents in the range 0.03 to 0.05% should be generally adequate for satisfactory spheroidal graphite formation.

1.4.4. Influence of trace elements

In the early stages of production of S.G.Iron, it has often been experienced that despite the presence of the desired amounts of residual magnesium contents, satisfactory spheroidal graphite structures were not being obtained. Detailed investigations have established that the degenerated graphite structures were attributable to the presence of certain types of trace elements present in certain types of pig irons and steels, which exercised a subversive influence on spheroidal graphite formation. While cerium reinforces the effect of magnesium and increases the tolerance range of the subversive elements, the preferred maximum limits for the different undesirable trace elements are as follows:

TABLE 1.2: Maximum limits for the different undesirable trace elements

Lead	0.002% max
Titanium	0.03% max
Antimony	0.004% max
Bismuth	0.002% max
Zirconium	0.1% max
Aluminium	0.15% max

1.4.5. Alloying elements

(a) **Nickel:** Nickel in small quantity is needed to promote pearlite like manganese but being a graphitiser does not give rise to carbides unlike manganese. Nickel content of 1.0 to 1.5% with suitably controlled manganese can give carbide free pearlitic as-cast structure. An optimum combination of tensile strength, fatigue strength, impact strength and war resistant properties, characterises such a structure. In normalised and quenched specimen, nickel promotes hardenability without losing the ferritic structure and UTS, yield strength of the specimen. However, nickel content in excess of 1.5% can retard the transformation of pearlite and thus prolong the annealing cycle to obtain fully ferritic structures, while in the predominantly pearlitic grades; higher nickel content might be permissible and in fact desirable particularly in heavy sections. In practice, it would be desirable to control the nickel content between 1.0 to 1.5%. An addition of 1% nickel will increase tensile and yield strengths by about 35 MPa with only a marginal reduction in the elongation.

(b) **Molybdenum:** Molybdenum promotes pearlite in as-cast S.G.Iron. It also retards annealing cycle. Molybdenum is mainly employed to enhance strength and hardness in heavy section castings. Tensile strength is increased by about 35 MPa for each 1% of Molybdenum in the ferritic grades while elongation is decreased by about 8%. Molybdenum increases response to heat treatment and is primarily added usually along with a certain amount of nickel for hardenability. Molybdenum and nickel together in controlled proportions develop an acicular structure, which lead to high tensile strength, toughness etc.

(c) **Copper:** Copper is used to a little extent as an alloy added prior to treatment with magnesium. It decreases ferrite and quite competent to stabilize pearlite. Copper does retard annealing, particularly at lower silicon contents. Copper decreases impact resistance and raises the transition temperature. It affects formation of spheroidal graphite formation.

(d) **Chromium:** Chromium is very potent carbide and pearlite stabilizer and is particularly detrimental for developing as-cast ferritic structure. On the other hand chromium is occasionally added for wear resistance and for hardness in heavy section castings.

Chromium increases oxidation and corrosion resistance in the austenitic S.G. Irons. Silicon is employed with chromium to reduce growth and to increase strength. [6]

1.5. MICROSTUCTURE

Engineering applications of cast iron have been traditionally based upon gray (Flake graphite) irons providing a range of tensile strengths between about 150 N/mm2 and 400 N/mm2 with recommended design stresses in tensile applications of 0.25×Tensile strength. Despite their limited strength gray irons provided very useful combinations of properties, which have ensured there wide continuing use. In fact gray irons still account for nearly 70 % of all iron castings produced. In contrast ductile irons have tensile strengths ranging from 350 to 1500N/mm2 with good elongation and high toughness. They now account for about 25 % of iron casting production serving in safety critical applications where they have replaced steel casting, forging and fabrications with technical and cost advantage.[17]

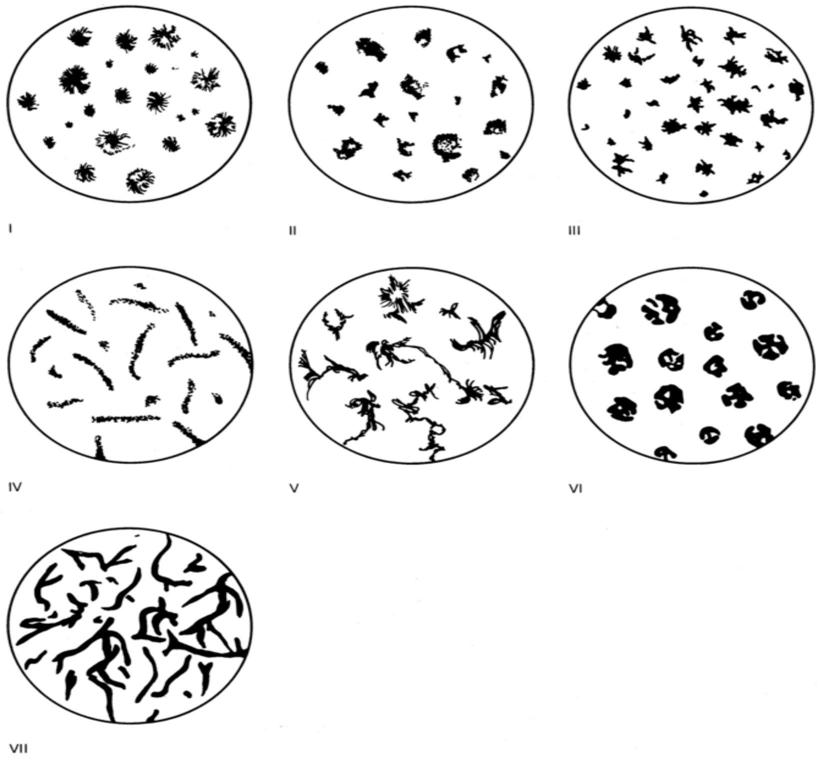


Fig.1.1: Typical shapes of graphite from ASTM A 247 standard. I) spheroidal graphite, II) imperfect spheroidal graphite, III) temper graphite, IV) compacted graphite, V) crab graphite, VI) exploded graphite, VII) flake graphite

Ductile iron and gray iron differs in morphology of graphite present in the structure. Ductile iron has graphite in the form of spherical nodules which is achieved by suitable treatments on the melt. The major micro structural constituents of ductile iron are: the chemical and morphological forms taken by carbon, and the continuous metal matrix in which the carbon and/or carbides are dispersed [17].

The following important micro-structural components are found in ductile iron:

Graphite

This is the stable form of pure carbon in cast iron. Its important physical properties are low density, low hardness and high thermal conductivity and lubricant property. Graphite shape, which can range from flake to spherical, plays a significant role in determining the mechanical properties of cast irons. Graphite flakes act like cracks in the iron matrix, while graphite spheroids act like "crack arresters", giving the respective irons dramatically different mechanical properties [18].

Carbide:

Carbide, or cementite, is an extremely hard, brittle compound of carbon with either iron or strong carbide forming elements, such as Chromium, Vanadium or Molybdenum. Massive carbides increase the wear resistance of cast iron, but make it brittle and very difficult to machine. Dispersed carbides in either lamellar or spherical forms play in important role in providing strength and wear resistance in as-cast pearlitic and heat-treated irons.

Ferrite

This is the purest in phase in a cast iron. In conventional Ductile iron ferrite produces lower strength and hardness, but high ductility and toughness. In Austempered Ductile Iron (ADI), extremely fine-grained acicular ferrite provides an exceptional combination of high strength with good ductility and toughness.

Bainite

When austenite is cooled to large supersaturations below the nose of the pearlite transformation curve, a new eutectoid product called bainite is produced. Like pearlite, bainite is mixture of ferrite and carbide, but it is by microstructural behaviour quite distinct from pearlite and can be characterized by its own C curve on a TTT diagram. In plain carbon steels this curve overlaps with the pearlite curve so that at temperatures around 500°C both pearlite and bainite form competitively.

In the case of pearlite the cementite and ferrite have no specific orientation relationship to the austenite grain in which they growing, whereas the cementite and ferrite in bainite do have an orientation relationship with the grain in which they are growing. Bainite is a mixture of ferrite and carbide, which is produced by alloying or heat treatment.

Pearlite

When austenite containing about 0.8wt% Carbon is cooled below the A_1 temperature (710°C) it becomes simultaneously supersaturated with respect to ferrite and cementite and a eutectoid transformation results, i.e. $\gamma \longrightarrow \alpha + Fe_3C$.In the case of Fe-C alloys the resultant microstructure comprises lamellae, or sheets, of cementite embedded in ferrite. This is known as pearlite. Both cementite and ferrite form direly in contact with the austenite. Pearlite nodules nucleate on grain boundaries and grow with a roughly constant radial velocity into the surrounding austenite grains. At small undercooling below A_1 the number of pearlite nodules that nucleate is relatively small, and the nodules can grow as hemispheres or spheres without interfering with each other. At larger undercooling the nucleation rate is much higher and site saturation occurs, that is all boundaries become quickly covered with nodules which grow together forming layers of pearlite outlining the prior austenite grain boundaries. A common constituents of cast irons, pearlite provides a combination of higher strength and with a corresponding reduction in ductility which meets the requirements of main engineering applications.

Martensite

Martensite is a supersaturated solid solution of carbon in iron produced by rapid cooling. In the untempered condition it is very hard and brittle. Martensite is normally "tempered"- heat treated to reduce its carbon by the precipitation of carbides-to provide a controlled combination of high strength and wear resistance.

Austenite

Normally, a high temperature phase consisting of carbon dissolved in iron, it can exist at room temperature in austenitic and austempered cast irons, in austenitic irons, austenite is stabilized by nickel in the range 18-36%. In austmepred iron, austenite is produced by a combination of rapid cooling which suppresses the formation of pearlite and the super-saturation of carbon during austempering, which depresses the start of the austenite-to-martensite transformation far below room temperature. In austenitic irons, the austenite matrix provides ductility and toughness at all temperatures, corrosion resistance and good high temperature properties, especially under thermal cycling conditions. In austempered Ductile Iron stabilized austenite, in volume fractions up to 40 % in lower strength grades, improves toughness and ductility and response to surface treatment such fillet rolling.

1.6. Family of Ductile Iron

With a high percentage of graphite nodules present in the structure, mechanical properties are determined by the Ductile Iron matrix. The importance of matrix in controlling mechanical properties is emphasized by the use of matrix names designate the following types of ductile iron.

(a)Austenitic Ductile Iron

Alloyed to produce and austenitic matrix, this ductile iron offers good corrosion and oxidation resistance, good magnetic properties, and good strength and dimensional stability at elevated temperatures.

(b) Ferritic Ductile Iron

Graphite spheroids in a matrix of ferrite provide and iron with good ductility and resistance and with a tensile and yield strength equivalent to a low carbon steel. Ferritic Ductile Iron cab be produced “as-cast’ but may be given and annealing heat treatment to assure maximum ductility and low temperature toughness.

(c) Ferritic Pearlitic Ductile Iron

These are the most common grade of Ductile Iron and are normally produced in the “as cast condition. The graphite spheroids are in a matrix containing both ferrite and pearlite. Properties are intermediate between ferritic and pearlitic grades, with good machinability and low production costs.

(d) Pearlitic Ductile iron

Graphite spheroids in a matrix of pearlite result in an iron with high strength, good wear resistance, and moderate ductility and impact resistance. Machinability is also superior to steels of comparable physical properties. The preceding three types of ductile iron are the most common and are usually used in the as-cast condition, but Ductile iron can be also be alloyed and/or heat treated to provide the following grades for a wide variety of additional applications.

(e) Martensitic Ductile iron

Using sufficient alloy additions to prevent pearlite formations, and a quench-and-temper heat treatment produces this type of ductile iron. The resultant tempered martensite matrix develops very high strength and war resistance but with lower levels of ductility and toughness.

(f) Bainitic Ductile iron

This grade can be obtained through alloying and/or by heat treatment to produce a hard, wear resistant material.

(g) Austempered Ductile iron

ADI, the most recent addition to the Ductile Iron family, is a sub-group of ductile irons produced by giving conventional Ductile iron a special austempering heat treatment. Nearly twice as strong as pearlitic ductile Iron, ADI still retains high elongation and toughness. This combination provides a material with superior wear resistance and fatigue strength.

1.7. Production of Ductile Iron

Ductile iron, also known as Spheroidal Graphite (S.G.) iron or nodular iron is made by treating liquid iron of suitable composition with magnesium before casting. This promotes the precipitation of graphite in the form of discrete nodule instead of interconnected flakes. The nodular iron so formed has high ductility, allowing casting to be used in critical applications. Such as: Crankshafts, steering knuckles, differential carriers, brake callipers, hubs, Brackets, valves, water pipes, pipe fittings and many others.

Ductile iron production accounts for about 40% of all iron castings and is still growing. While a number of elements, such as cerium, calcium and lithium are known to develop nodular graphite structures in cast iron, magnesium treatment is always used in practice. The base iron is typically:

TABLE 1.3: Determinants of base iron

%C	%Si	%Mn	%S	%P
3.7	2.5	0.3	0.01	0.01

Having high carbon equivalent value (CEV) and very low sulphur, sufficient magnesium is added to the liquid iron to give a residual magnesium content of about 0.04%, the iron is inoculated and cast.

1.8. Properties of S.G.Iron

A number of properties such as mechanical, physical and service properties are of importance in assessing a material's suitability for any application. The mechanical properties of interest are tensile strength, proof stress, elongation, hardness, impact strength, elastic modulus, fatigue strength and notch-sensitivity while the physical properties of interest are damping capacity, machinability and thermal conductivity. The service properties generally involved are wear resistance, heat resistance and corrosion resistance. Each of these properties will be surveyed below briefly.

1.8.1. Mechanical Properties

Because of the spheroidal nature of the graphite, the tensile properties, hardness and impact strength of S.G.Iron approach nearly those of the matrix. The as-cast matrix consists of varying proportions of pearlite and ferrite and also cementite depending on the metal composition and the rate of cooling or in other words, section thickness of the casting. The elimination of carbides, changing the proportions of pearlite and ferrite and refining of pearlite can be achieved by different types of heat treatment such as quenching and tempering, normalising and tempering, normalising, controlled cooling, full annealing and sub-critical annealing. The 0.2% proof stress will usually vary between 0.7 to 0.6 of the tensile strength as strength increases except in the hardened condition, where it will be about 0.75 of the tensile strength. The proportions of the different constituents of the matrix are also affected by the amount and types of alloying element present. The matrix strength is also improved by alloy additions such as nickel and molybdenum in particular.

Special matrix structures such as bainitic, martensitic and austenitic, with considerably altered mechanical properties, can be developed through alloy additions.

Unlike in the case of grey cast iron and as in the case of steel, the elastic modulus of S.G.Iron does not vary with strength and is about 1.76×10^6MPa.

The fatigue strength of S.G.Iron bears a certain ratio to its tensile strength, but the ratio varies as in the case of steel as well, depending upon the strength level. The ratio which is about 0.45 in the case of the more ductile ferritic grades falls to about 0.40 as the strength goes up to about 80 Kg/mm2 and still less with a further rise in strength. The fatigue properties of a material are considerably influenced by the notch-sensitivity factor, which is the ratio of the unnotched and notched fatigue strengths.

1.8.2. Physical Properties

Although the spheroidal nature of the graphite decreases damping capacity compared to flak graphite grey cast iron, it is still significantly higher compared to steel. The camping capacities of steel, S.G.Iron and flak graphite grey cast iron may be taken to be in the ratio 1:1.8:5. The relatively higher damping capacity of S.G.Iron compared to steel is an advantage in certain application as it causes less tool chatter and noise emission is gearing.

Like flake graphite grey cast iron, the machinability of S.G.Iron is also good, being able to the same for the same hardness. For the same strength, S.G.Iron is the most readily machinable ferrous material. Machinability decreases as the matrix changes from more of ferrite to more of pearlite. Presence of carbides particularly impairs machinability.

Bother steel and cast iron have relatively higher thermal conductivity compared to any of the S.G.Iron types. The thermal conductivity of the austenitic types of S.G.Iron is even less. The ferritic types of S.G.Iron have relatively higher thermal conductivity compared to the pearlitic types. Thermal conductivity is a vital property where rapid heat dissipation is called for in a component and it must be regarded that S.G.Iron is placed somewhat disadvantageously in respect of this property.

1.8.3. Service Properties

A service property that has led to the extensive use of S.G.Iron in many applications is its outstanding wear resistance. Crankshafts, Metal working rolls, Punch dies, Sheet Metal dies and Sheaves are representative examples. Compared to steel, S.G.Iron at equal hardness is markebly superior. Because of the spheroidal graphite, galling and scuffing in the machinery parts are obviated. The wear resistance property has been a major factor for the acceptance of S.G.Iron as a material for gears.

In most cases, the corrosion resistance of S.G.Iron is similar to that of grey cast iron but in some it shows decided improvement. Compared with carbon steel, it has good resistance to attack by aggressive atmosphere, sea water, alkalis and some weak acids, Together with its superior mechanical properties, this has prompted the use of S.G.Iron in the chemical and petroleum industries and for marine applications. Corrosion resistance is enhanced by an austenitic matrix.

At high temperatures, flake graphite cast iron suffers structural changes and gas penetration and the consequent internal oxidation result in physical growth. S.G.Iron is dimensionally much more stable at high temperatures, since the graphite spheroids are isolated from each other and do not provide paths for penetration of gases, as do the networks of graphite flakes in ordinary cast iron. Surface oxidation of S.G.Iron is also appreciably less. These twin advantages account for the superiority of this iron for such applications as furnace doors, Refractory tile hanger for furnace walls and general furnace castings. The creep strength of S.G.Iron is notably good and renders the material suitable for steam plant and other medium temperature work. Flake graphite iron is susceptible to fracture under thermal shock; by contrast, S.G.Iron has very high resistance. A drastic test of this property is provided by the quenching of an S.G.Iron refinery valve which had been bolted to a steel pipe form a temperature of 730° C by means of a fire hose. In this test, the steel fittings buckled, the steel sheared, but the S.G.Iron valve itself was undamaged [6].

1.9. HEAT TREATMENT

1.9.1. Annealing

The purpose of annealing heat treatment in the case of SG iron castings is to have maximum ductility and good machinability. After annealing, the microstructure consists of graphite nodules in ferrite matrix.

There are three methods of annealing: (i) Heating the castings to 900-950°C and holding for 1 hour plus 1 hour per 25 mm of section thickness of casting. For heavy casting, the holding time may be up to 8 hours. After this, casting is cooled to 690°C and is kept at this temperature for 5 hours plus 1 hour per 25 mm of section thickness. (ii) In this case, the casting, after being held for 1 hour for 1 hour at 900-955°C, is furnace cooled to 650°C. Cooling rate between 790°C and 650°C should not exceed 20°C/hour. (iii) In both cases (i) and (ii), the matrix obtained is fully ferritic. When impact strength is not of significance, carbides can be tolerated in casting. Under such conditions, castings are heated up to 700°C and held there for 5 hours plus 1 hour per 25mm of section thickness. After this, castings are cooled to 590°C in furnace. This is a subcritical annealing treatment for SG iron. For superior machinability, Mn, P and alloying elements such Cr, Ni and Mo should be as low as possible. There are carbide formers. Of these, chromium carbide takes longest time to decompose at 925°C.

1.9.2. Hardening and Tempering

For hardening of SG iron, castings are austenitized at 845-925°C. This is followed by oil quenching. To reduce stresses, oil is preferred as quenchant. Water and brine can also be used in place of oil as quenchant for castings with simple shapes. Castings which have complex shape are quenched in hot oil maintained at 80-100°C to avoid quench cracks. Immediately after quenching, castings are tempered in the range 300-600°C for 1 hour plus 1 hour per 25mmm section thickness. This is to minimize quenching stresses. Tensile strength of quenched SG iron ranges from 700 MPa to 1000 MPa. The yield strength of such heat treated SG iron varies from 540 MPa to 880MPa, with elongation of 10-12 percent and hardness of 215-320 BHN. The Microstructure of quenched SG iron reveals the martensitic matrix.

1.9.3. Normalizing

The purpose of normalizing is to improve the tensile properties of SG iron. For normalizing, SG iron is heated up to 870-940°C and soaked there for 1 hour. Temperature and soaking time vary with composition, especially with silicon and chromium contents. Normalizing is commonly followed by tempering to achieve the required hardness and to relieve stresses developed during air cooling because of different section sizes of castings. Tempering is carried out at 510-620°C for 1 hour. Tempering after normalizing is also adopted to improve toughness and impact resistance in addition to tensile strength. For these castings, after air cooling from 870°C to 940°C, are reheated to 425-650°C and soaked for 1 hour. Normally, the microstructure after normalizing consists of fine pearlite and globular graphite. Allying additions such as nicked, molybdenum and additional manganese help to develop a fully pearlitic structure after normalizing. Martensitic structure is obtained in the case of alloyed castings which are not heavy. Alloying additions increase hardenability.

1.9.4. Austempering

To achieve the full potential of SG iron, austempering heat treatment is adopted. It is possible to achieve much higher ranges of tensile strength and elongation by adopting austempering treatment for SG irons.

The process as such is very simple. The first stage consists of soaking the castings at the austenitizing temperature of 850-950°C. The austenitized castings are then quickly transferred to a liquid bath maintained in the temperature range of 235-425°C. The transformation is allowed to proceed for a period of up to 4 hours when austenite transforms to bainite. The castings are finally cooled to room temperature after transformation.

By adopting austempering instead of conventional hardening and tempering treatment for SG iron, the requirement of alloy content is reduced for the same strength level.

It is possible to achieve various combinations of high strength, high hardness, limited ductility or lower strength, lower hardness, high ductility by varying the temperature of austempering. The austempered ductile irons have good machinability. It is comparable with that of other ductile irons or low alloy steels. However, in such cases where higher hardness is armed at by heat treatment,, machining should be carried out prior to the heat treatment.

These materials have higher wear resistance than other ductile irons due to the presence of bainite. It may even be better than some steels. However, it has limited wear resistance as compared to white cast iron. For the same reason, the fatigue properties are superior to other ductile irons.

TABLE 1.4: **Comparative Properties of Austempered Ductile Irons and Other Materials**

Material	Tensile strength (MPa)	Yield strength (MPa)	Hardness (BHN)	Elongation (percent)
	Ductile Irons			
Pearlitic and ferritic	400-960	250-610	130-300	28-3
Austempered	800-1600	600-1400	250-480	16-1
Hardened and tempered	600-1300	500-1100	300-400	5-1
	Steels			
Hardened and tempered	700-1800	450-1450	210-510	25-8

The impact values of austempered ductile irons are much higher than pearlitic ductile irons. They are in fact comparable to those of ferritic ductile irons. The transition of ductile to brittle fracture occurs at a lower temperature range in austempered ductile irons than in pearlitic ductile irons. Current studies on fracture toughness indicate that austempered ductile irons have remarkably higher range of values than those of other ductile cast irons.

Austempered ductile irons have great potentiality for use as gears. The advantageous property of high damping capacity of cast iron will be useful to minimize vibrations and transmission noise. Advantages of austempered ductile irons include high strength and ductility, wear resistance and toughness and better machinability, higher damping capacity and reduced weight in comparison with steel.

TABLE 1.5: **Possible Engineering Applications of Austempered Ductile Irons**

Automobiles	Crankshafts
	Camshafts
	Steering knuckles
	Suspension components
Pumps and Compressors	Bodies
	Crankshafts
	Drive shafts
Railways	Couplings
Agriculture	Undercarriage parts
	Constructional equipment

Applications of austempered ductile irons based on their wear resistance will include pump castings and impellers of sludge handling equipments, plough shears in agriculture and forestry, friction blocks and locomotive wheels, conveyor rollers and blades and shredders in general machinery.

1.10. Application of SG iron

The application of S.G.Iron is numerous and can be found almost in every branch of Industry. While several of the existing applications involve substitution of other materials, a stage has been reached in the development of the material to merit serious attention to design components to suit its own properties to derive full economic and technical advantages from the use of the material. The characteristic properties of S.G.Iron that merit serious attention to design components to suit its own properties to derive full economic and technical advantages from the use of the material. The characteristic properties of S.G. Iron that merit the attention of Designers may be summarised as follows:-

(i) Excellent fluidity enabling intricate shapes to be cast readily.
(ii) Feasibility of producing spheroidal graphite structures in an almost unlimited range of section sizes with very little falling off in mechanical properties.
(iii) Feasibility of developing, by suitable heat treatment and alloying, tensile strengths of over 90 Kg/mm2 with limited shock-resistance over elongations of over 15-20% coupled with a tensile strength of nearly 50 Kg/mm2.
(iv) Good wear-resistance which can be further improved by a surface-hardening treatment.
(v) Corrosion resistant properties, superior to those of low carbon cast steels.
(vi) Resistance to growth and scaling at elevated temperatures, much superior to that of flake graphite grey cast irons.

By virtue of its versatile properties, S.G.Iron has replaced not only the other types of ferrous castings but also steel forgings in many applications. Spun pipes, metal working rolls and automobile components may be regarded as the main tonnage markets for S.G.Iron in general.

Pipe is major application of cast iron and cast iron pipe is used extensively for the transport of gas, water, etc. in cities. Ordinary cast iron suffers from the disadvantage that under heavy traffic conditions or where soil subsidence takes place, the pipe is liable to fracture. The superior strength and toughness of S.G.Iron has, therefore, led to adoption as material for service pipes in many cities throughout the world.

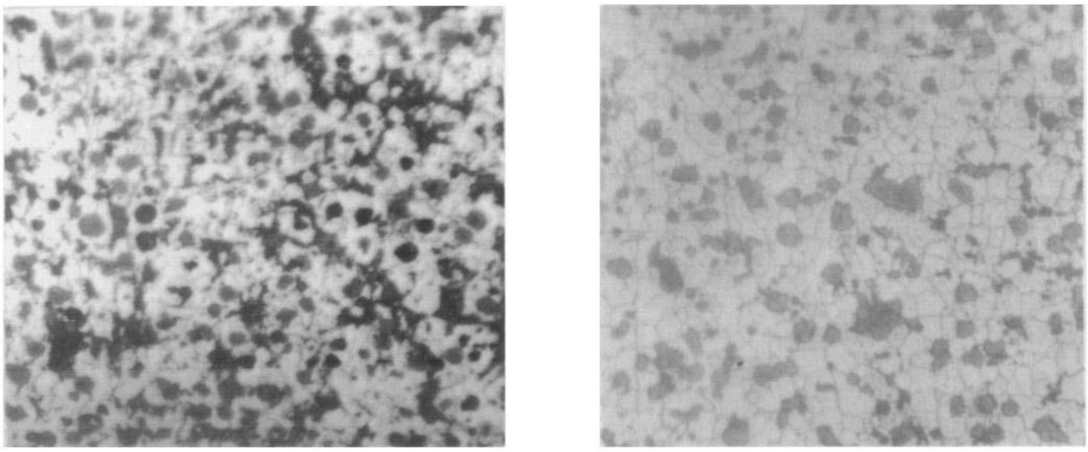
A currently major application of S.G.Iron in India is railway bearing shells. The original material specification for the item is phosphor bronze. S.G.Iron has been successfully established for the item, involving a demand of nearly 3000 tonnes per annum, after extended and satisfactory trials. The enormous saving in cast and foreign exchange by the changeover to S.G.Iron from phosphor bronze for the large quantities involved needs no elaboration.

One of the technically interesting applications of S.G.Iron is crankshafts of air and refrigeration compressors and petrol and diesel engines.

Throughout the world, the automobile industry constitutes a vital field of application for S.G.Iron. It is expected that before long this will be the position even in regard to the Indian automobile industry. Some of these automobile applications are proposed to be referred to in some details with particular reference to their manufacture in the author’s foundry.

Chapter 2
Literature Review

Sheng Da and Zheng Yongping[7] says Continuous casting is better procedure in comparision to centrifugal casting. By adding REMgSiFe, continuous casting can be obtained. His paper throws light on rare earth ductile iron pipes and how elements change the structure and properties of the iron. Untreated ductile iron pipe has strength of 515-485MPa while 2.5-2.7% elongation. By heat treatment this value changes remarkably in case of % elongation by 7-16% at the same time by retaining strength of 440-425MPa. The phases found in ductile iron pipes to be ferrite or pearlite or ferrite plus pearlite. This developed iron pipes is being used in different fields of China. The cost incurred is less in producing continuous casting rare earth ductile iron pipes unlike in centrifugal path. Slag inclusions, gas hole and shrink marks are main defects in continuous casting rare earth ductile iron pipe. Slag inclusions can be decreased by controlling metallurgical quality and reducing spheroiditc agent in liquid cast iron i.e. controlling the Mg, Ce, Al, S, Si, Mn, Ti, O, Cu, Cr, Mo, Ni and P content will lead to lesser slag inclusions.



Untreated Treated

Fig.2.1: Optical images of (a) Untreated and (b) Treated rare earth ductile cast iron

These figures clearly reveal that percentage of ferrite in treated cast iron increased which leads to increased value of percentage elongation (7-16%).

Konoplyuk S, Abe T, Uchimoto. T, Takagi. T, Kurosawa [8] has found a new method to evaluate properties of ductile cast iron and is called Eddy current method. A good correlation of hardness and tensile properties are established by measuring responses of eddy current signals from FCD 450-600 grades cast iron. Whereas ultrasonic method of measuring hardness could not give the expected results. This non-destructive eddy current method predict matrix dependent properties assessment such as hardness and tensile properties of ductile cast irons, with large numbers of heat treated cast irons and with different chemical compositions, unlike ultrasonic method which fails to do it.

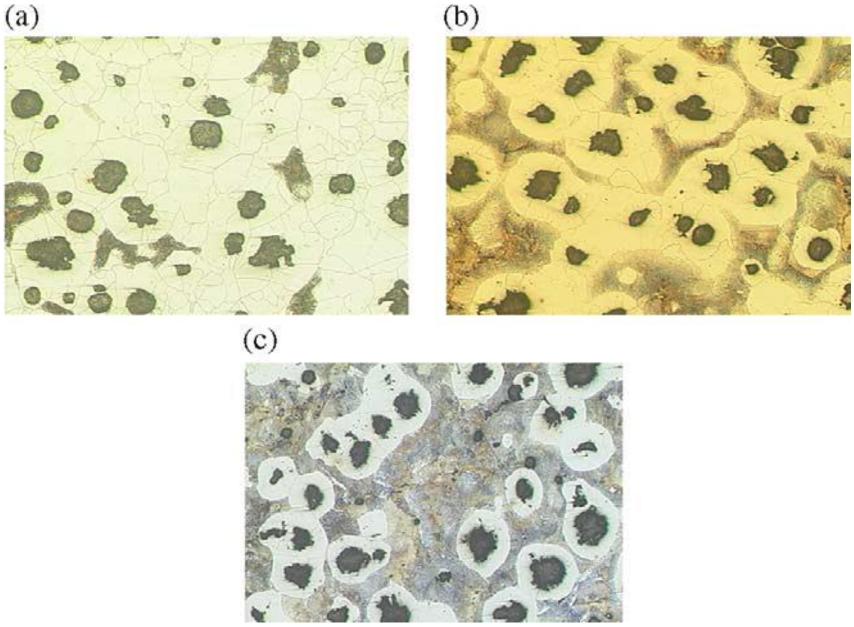


Fig.2.2 :(a) Ferrite, (b) Pearlite (Bull eye), and (c) Intermediate optical images of ductile iron

These three images shows the types of iron studied under this Eddy current response method of assessing mechanical properties.

Pongsak Chaengkham and Panya Srichandr [9] have studied ductile iron billets made from continuously cast horizontal casting machine and compare the mechanical properties of it to the cast iron made from conventional sand castings. The overall quality of continuously cast ductile iron found to be acceptable. The carbide content of ductile irons, made from both the traditional sand casting and the castings made from horizontal casting machine, are compared. The microstructure of as-cast iron may have eutectic carbide, chunky carbide or graphite nodules in pearlitic matrix. Nodularity of as-cast iron depends on holding time of melt iron, which has been inoculated and treated with elements like Mg, Ce for nodularity, followed by casting. Nodularity, percentage of spherical nature of graphite nodules, has been found to be decreasing as the melt held for 6 min or longer. This decreasing has effect on tensile strength and percentage elongation of the cast. The mechanical properties like tensile strength of 410-550 MPa and % elongation of 2.5-4.0%, 520-550HB hardness, was found. The surface texture was also found to be good enough though some marks were present.

Again, S Konoplyuk[10] has found a method to estimate pearlite content in ductile cast irons by eddy current method. By this method graphite particle’s or pearlite’s size and its amount present in the matrix can be predicted.

M.A. Shaker[11] in his paper throws the light on graphite shape in different types of cast iron like gray, compacted and ductile cast iron. The mechanical properties because of graphite shape in gray cast iron is completely different to both compacted or ductile cast iron. This paper studied the outcomes of changing tempering temperature and the different shape of graphite in various types of cast irons. The results of this study is clearly saying that controlled graphite-shape and nodularization percentage are basic properties parameter of cast iron. Workability limits are found to be improved by heat treatment, hardening and tempering. Increasing nodularization percentage increases workability.

R.C.Dommarco, M.E.Sousa, J.A.Thin Sikora[12] To improve strength to weight ratio, wall ductile iron is being used. Thin walled iron has increased nodule count and the nodule count has tremendous effect on the abrasion resistance of different matrix microstructure. The nodule count for this paper has been taken between 250 to 2000 nod/mm^2 and thickness of test samples is form 1.5 to 25mm. To achieve fully martensitic, ferritic, pearlitic and ausferrtic microstructure, the sample are heat treated. In all heat treated samples, it is found that on increasing nodule count abrasion resistance decreases. As per ASTM G 65 standard, abrasion resistance of different matrix with different nodule count has been evaluated. The result indicates that as the nodule count in a particular matrix increases, its wear rate increases which lead to lower abrasion resistance. So at the end it concluded that as the concentration of nodule increases, wear resistance decreases.

M Rashidi Ali, M Moshrefi-Torbati[13] This paper deals with the response of ductile iron to the tempering time and temperature. Impact strength and ductility increases suddenly with tempering between 450 to 500°C. Initially UTS decreases with increasing tempering temperatures, between 400 to 500°C remains constant and then decreases again. The effect of tempering time is also as important as the tempering temperature and effect as follow: up to 120 min, the UTS and Yield stress decreases on increasing periods, impact strength increases up to 90 min and ductility increases to 120 minutes period. Effect of tempering temperature and tempering period on ductile cast iron has been shown graphically as below,

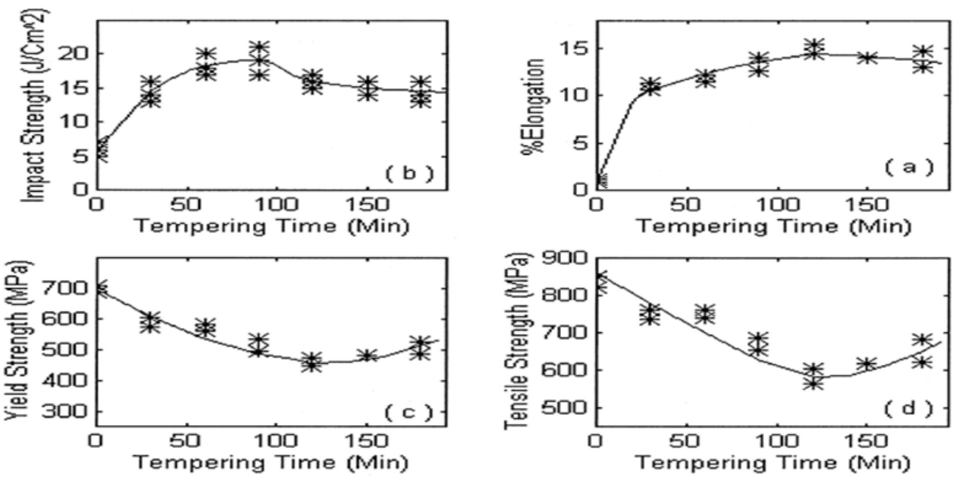


Fig.2.3: Effect of tempering temperature and tempering period on ductile iron.

D.C.Wen, T.S.Lei[14] The effect of tempering on mechanical properties of ADI(austempered ductile iron) is discussed in this paper. To get ADI, the parent cast iron is heat treated which leads to ausferrite and tempered martensite matrix structure. With increasing martensite concentration, mechanical properties improve. Here heat treatment is so done to achieve both ausferrite and tempered martensite phases in the matrix. Austempering was followed by tempering at 473 K for 7.2 ks and brought to the room temperature. This mixture of heat treatment and phases give significant improvement in mechanical properties of ADI, specially tempering at 473 K in the last stage decrease the sensitivity of mechanical properties. Mechanical properties are shown to improve till increased martensite content. The results obtained are as follows:

- The martensite size increased with increasing austempering temperature and martensite content decreased. On increased martensite content the properties like ductility and toughness decreased
- Unwanted effect of martensite can be eliminated by following tempering at 473 K i.e. toughness and strength can be recovered by following tempering after austempering. This combined process could also optimize the mechanical properties up to great extent and shorten the austempering time.

- The mechanical properties increase after tempering because of the increased martensite content. This increased mechanical property has nothing to do with matrix of ausferrite and austempering temperature i.e. this is independent of morphology of ausferrite and austempering temperature.

R.Konečná, G.Nicoletto, L.Bubenko, S.Fintová[15] throws light on comparison of Isothermal Ductile Iron (IDI) and Austempered Ductile Iron(ADI). This paper says IDI is better option in relation to ADI in applications subjected to dynamic loading. Both irons have different metallic structure. The IDI results in high cycle fatigue strength, high fatigue crack growth rate and threshold K_{th}, the typical crack propagation mechanisms and the material heterogeneity in terms of local tensile tests. Metallographic analysis shows similar graphite nodule characteristics in both ADI and IDI, with different matrix structure. After investigating ADI and IDI, following results are found:

- Tensile strength of both the irons is recorded and it is found that high cycle fatigue strength of ADI is higher than IDI.
- Fatigue crack growth and threshold K_{th} of IDI is higher than ADI.
- Crack path is different in both the irons, ADI and IDI.
- Because of very fine microstructure of both the irons, fatigue fracture roughness is low in the both. Because of softer phase of IDI, roughness is high in comparison to ADI.

Uma Batra, Pankaj Tandon, Kulbir Kaur [16] Here three temperatures, 850, 900 and 950°C selected to austenising process of three different composition SG iron and their metallographic study, hardness and XRD test is carried out and found that optimum austenization time is maximum for ferritic and minimum for pearlitic matrix.

Austempered ductile irons (ADI) have outstanding mechanical properties and been major replacement for steel in wide ranges of industries, right from agricultural equipment to medical equipments and it has also a huge potential applications in almost all industries. As-cast material is heated to 850-950°C temperature range and kept there for 60-90 min, on the basis of dimension of the specimen selected, followed by quenching to 250-425°C and being hold there for isothermal transformation and at last the specimens are air or water quenched. The ferrite or ferrite plus pearlite or pearlite, transform to homogenized austenite, $\acute{Y}_{0.}$ There are two stages involved in austempering reaction, in the first stage austenite matrix transform to bainitic ferrite by rejection of carbon, by rejection of carbon by bainitc ferrite into rest austenite to form high carbon austenite. In second stage, the high carbon austenite formed in stage 1 transforms to bainitic ferrite and carbide. If the carbon content of austenite formed in stage 1 is high enough then it will be retained during cooling, however if the carbon content of austenite is not enough then it will transform to martensite during cooling. The carbon content of austenite formed in stage 1 depends on the carbon content of initial austenite, $\gamma_{0,}$ formed during austenitization and amount of carbon rejected into austenite during bainitic transformation in stage 1 of austempering increases with the decrease of carbon content of initial austenite, γ_0 (Darwish et al 1993). It can be seen from above discussion that carbon content of initial austenite, $\gamma_{0,}$ is most important element in the transformation of austenite.

The importance of the austenitization parameters i.e. temperature, time and the cast structure has been studied on ferritic, ferritic plus pearlitic and pearlitic ductile irons. Austenitizatin is promoted significantly when there is a high pearlitic content in the as cast matrix structure. Increasing the austenitization temperature accelerates the austenitization kinetics and increases the carbon solubility in the austenite phase. The optimum austenitization temperature and time is that at which austenite with maximum and uniform carbon has been achieved. At the austenitization temperature of 900C, the optimum austenitization time for Ni-Mo iron is 90 min, for Cu-iron 1 70 min and for cu-iron 11 60 min. The equilibrium carbon content of austenite is 0.67% for Ni-Mo iron, 0.81% for cu-iron 1 and 0.87% for Cu-iron 11.

Chapter 3

Experimental Procedure

- Specimen Preparation
- Heat Treatment
- Hardening and Tempering
- Normalizing
- Annealing
- Austempering
- Mechanical Testing
- Tension Test
- Vicker's Hardness
- Fractography and Metallographic Analysis
- XRD

3.1. Specimen Preparation

Tensile and impact specimens according to ASTM E8 and ASTM D256 standards respectively are machined from ductile cast iron test block brought from L&T Kansbahal, India. Composition of the test block is given in table 3.1.

Table 3.1

Elements	Wt%
C	3.61
Si	2.1
Mn	0.2
S	0.007
P	0.022
Cr	0.03
Ni	0.47
Mo	0.001
Cu	0.009
Mg	0.043
Ce	0.004
Fe	balance

3.2. Heat Treatment

3.2.1. Hardening and Tempering

Specimen is austenitized at 1000°C and kept there for 90 minutes. After austenitizing the specimen is quenched in mineral oil maintained to 100°C. After keeping at that temperature for 30 minutes specimen is tempered at 500°C for 120 minutes, followed by air cooling to room temperature.

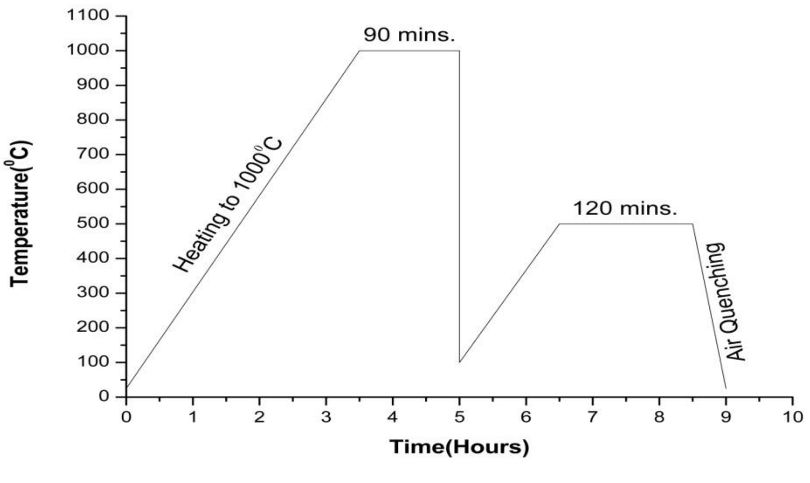


Fig.3.1: Diagram showing Hardening & Tempering heat treatment process.

3.2.2. Normalizing

Specimen is austenitized at 1000°C and kept there for 90 minutes. After austenitizing the specimen is air cooled to room temperature.

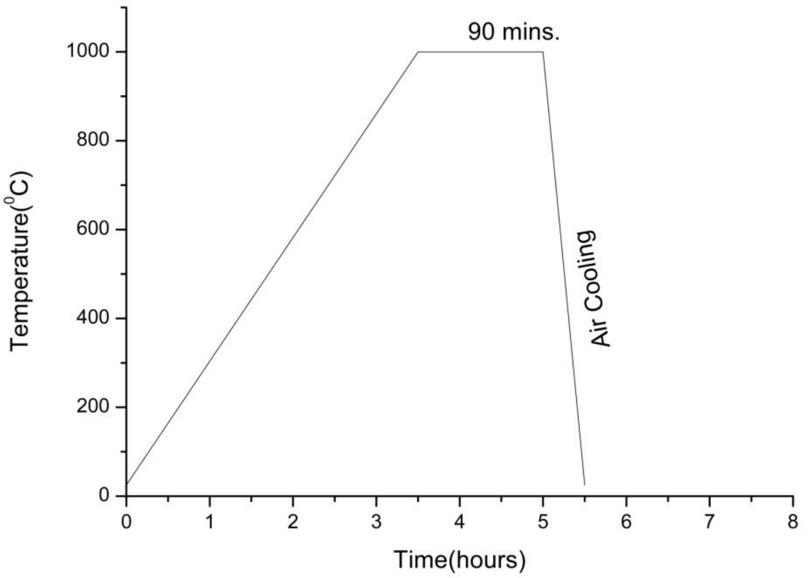


Fig.3.2: Diagram showing Normalizing treatments

3.2.3. Annealing

Specimen is austenitized at 1000°C and kept there for 90 minutes. After austenitizing the specimen is furnace cooled up to 700°C and kept there for 5 hr 30 minutes. After that specimen is cooled to room temperature in the furnace.

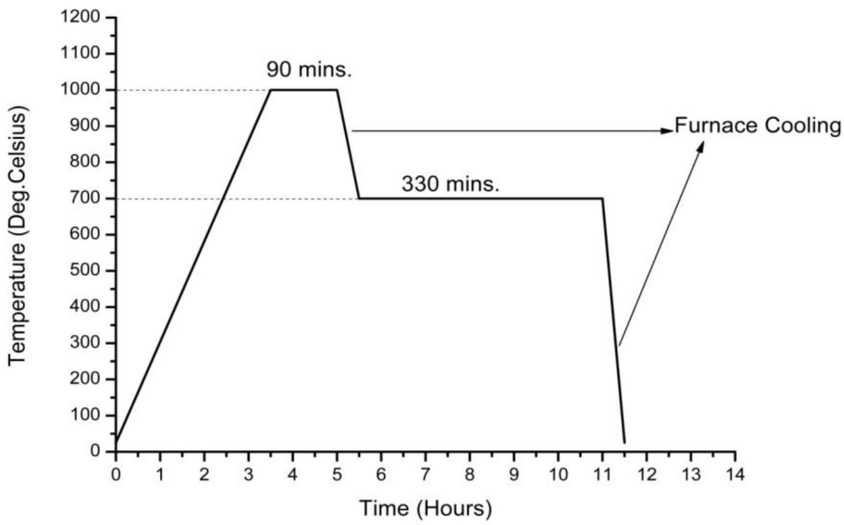


Fig.3.3: Diagram showing Annealing treatment

3.2.4. Austempering

In this heat treatment process the specimen is austenitized at 1000°C and kept there for 90 minutes. The specimen is quenched in salt bath which was prepared by mixing NaNO3 and KNO3 in 1:1 ratio at 500°C. The specimen in the salt bath is maintained at 500°C for 4 hours. After this the specimen is taken out and air cooled to the room temperature.

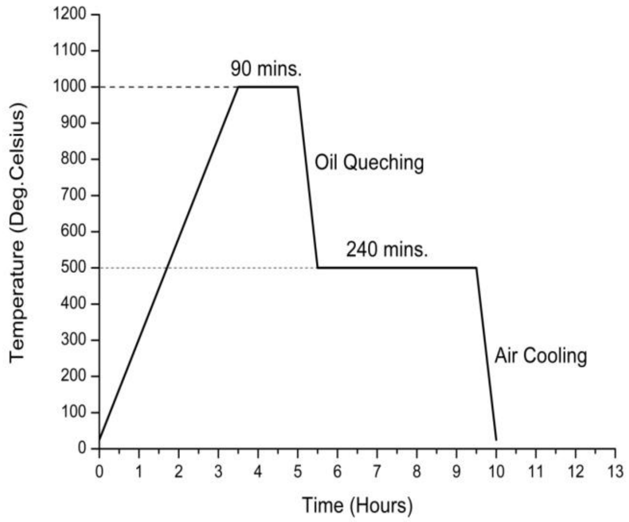


Fig.3.4: Austempering treatment has been shown in the above diagram.

3.3. Mechanical Testing

3.3.1. Tension Test

Tension test is carried on the specimen using INSTRON 1195 and Ultimate tensile strength (UTS), 0.2% YS, Elongation are recorded.

Fig.3.5: INSTRON 1195; Tension Test Equipment

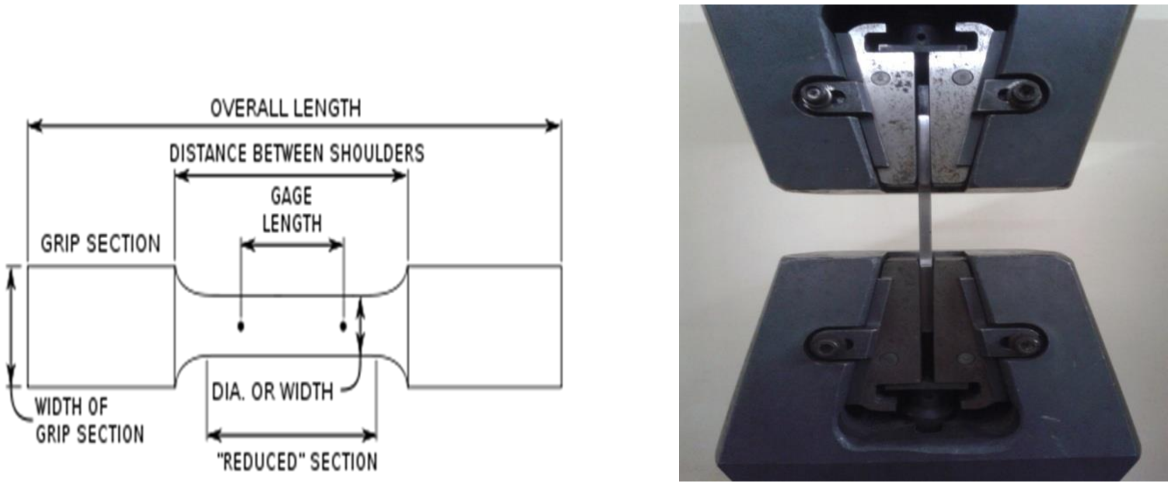


Fig.3.6: ASTM E8, Flat Sub-size Specimen

3.3.2. Vicker's Hardness

Hardness of as-cast and heat treated specimens were carried out using Vicker's Hardness Tester, dwell time of 10 sec. and load of 20 kg.

Fig.3.7: Vicker's Hardness Tester

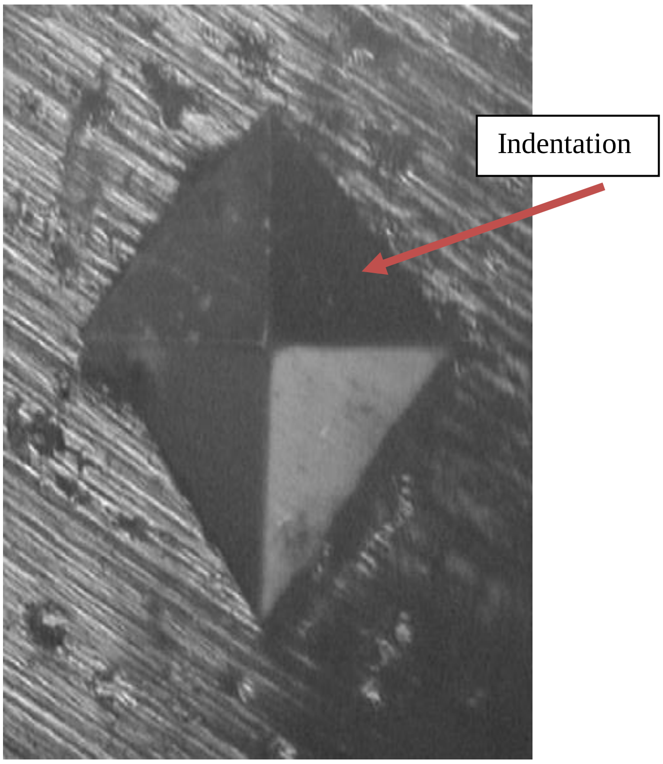


Fig.3.8: Specimen showing indentation

3.3.3. Impact Test

Impact test is carried out to know the impact strength of as-cast and heat treated specimens.

3.9 Impact Tester

3.4. Fractography & Metallographic Analysis

Fracture surfaces after tension test for respective heat treated and as-cast specimens are observed under Scanning Electron Microscope (SEM) make-JEOL, model-JSM 6480LV.The Factrographs of as-cast, tempered, normalized, annealed and austempered specimens were taken.

Metal Power Image Analyser was being used to study the microstructure of as-cast and heat-treated specimens. Prior to the metallographic study specimens were well polished using emery papers of different grades followed by cloth polishing with diamond paste. Specimens are etched with 2% nital solution after the polishing.

Fig.3.10: Scanning Electron Microscope (SEM) make-JEOL, model-JSM 6480LV

Fig.3.11: Metal Power Image Analyser with Optical Microscope

3.5. XRD

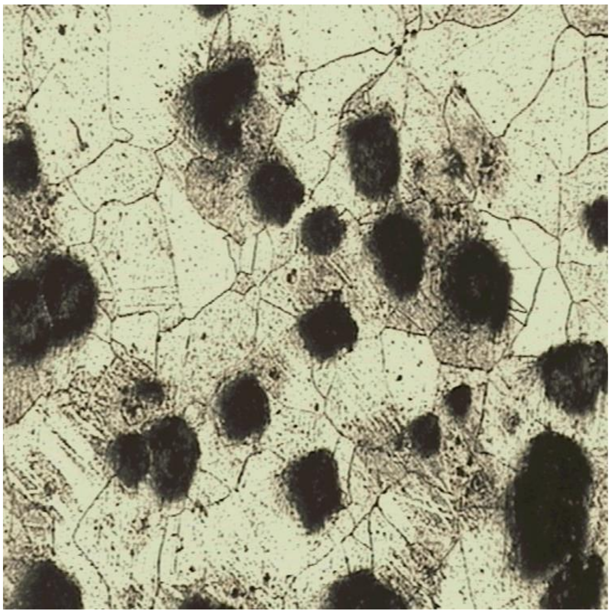
X-ray diffraction technique was used to determine crystal structure using X’pert PANalytical PPW3040/00. Step size of 2Ө per minute and 2Ө range of (30-90) ° were applied.

Fig.3.12: XRD machine; X’Pert PANalytical PPW3040/00

Chapter 4 Result and Discussion

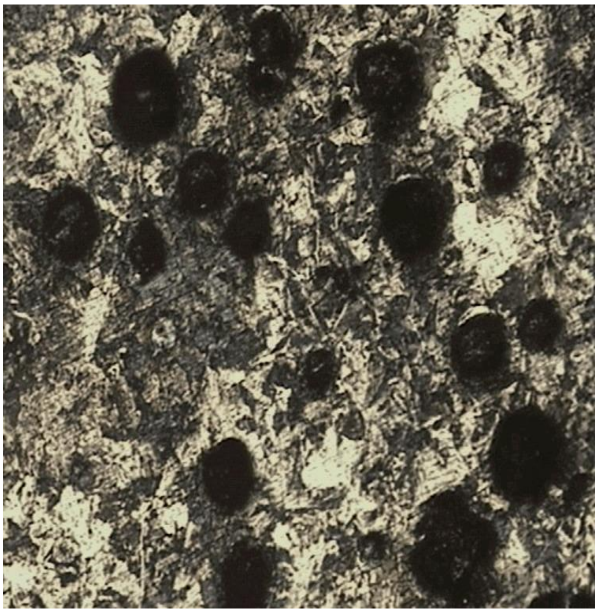
4.1. Metallographic Analysis

Metallographic images for as-cast and heat-treated specimens are presented in figure 4.1. Fully ferritic matrix with spherical graphite nodules are observed for annealed and as-cast specimens. Ferritic/pearlitic microstructure was obtained for normalized specimen where as hardened and tempered specimen was observed to have tempered martensite. On the other hand austempered specimen appeared to have ausferritic matrix. The quantitative metallographic analysis had carried out on each of the treated as well as on as-cast specimen. Graphite area fraction, nodularity, nodule count were obtained from quantitative analysis are presented in table 4.1. The nodularity for each of the specimen was found to be more than 90 percent. Least value of 92.625% obtained for as-cast specimen whereas a maximum value of 98.092 percent was obtained for annealed and normalized specimens. Although there is no significant variation in nodularity but significant variation was observed in nodule count for respective specimens. The least value for nodule count 991 was obtained for hardened and tempered specimen whereas maximum value of 2542 was obtained for austempered specimen. Nodule count for as-cast, normalized & annealed specimen are in between i.e. 1217, 1763 & 1943 respectively. Similarly variation in graphite area fraction was also observed with maximum value of 32.072% for normalized specimen and minimum value of 21.499% for austempered specimen. These variations in nodule count and graphite area fraction can be attributed to the fact that the carbon atoms get diffused into different matrices like pearlite, tempered martensite & ausferrite during respective heat treatment processes. However, although both as-cast & annealed specimens have fully ferritic microstructure, the change in graphite area fraction i.e. from 28.910% to 25.168% for respective specimens is clearly visible in change in percentage nodularity which increases from 93.625% for as-cast specimen to 98.092% for annealed specimen. Except normalizing heat treatment process, there is decrease in graphite area fraction for each of the heat treatment process. There is a decrement of 25.63 % in graphite area fraction for austempering heat treatment process whereas an increment of 9.85% graphite area fraction for normalizing heat treatment process was observed as compared to as-cast condition.

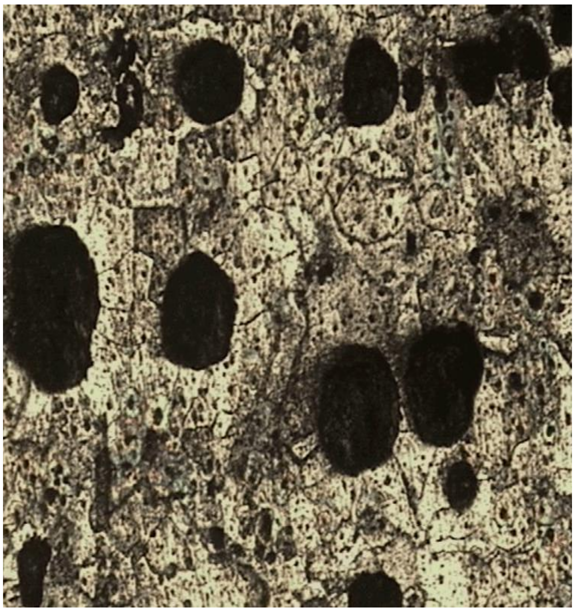


(1) As-Cast

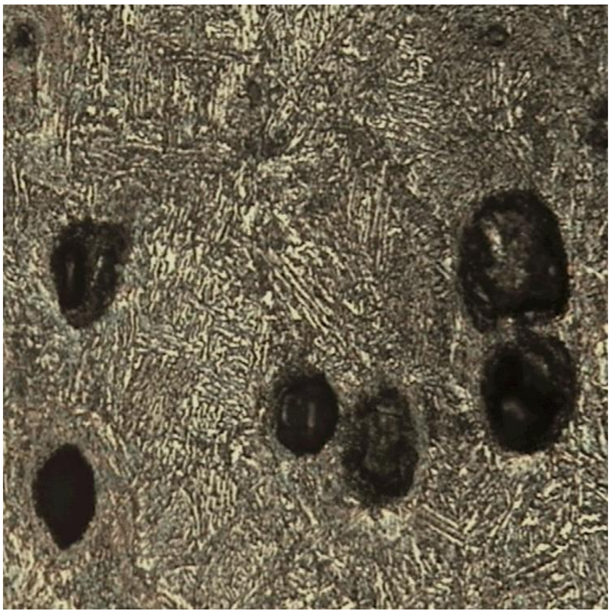
(2) Hardened & Tempered



(3) Normalized



(4) Annealed



(5) Austempered

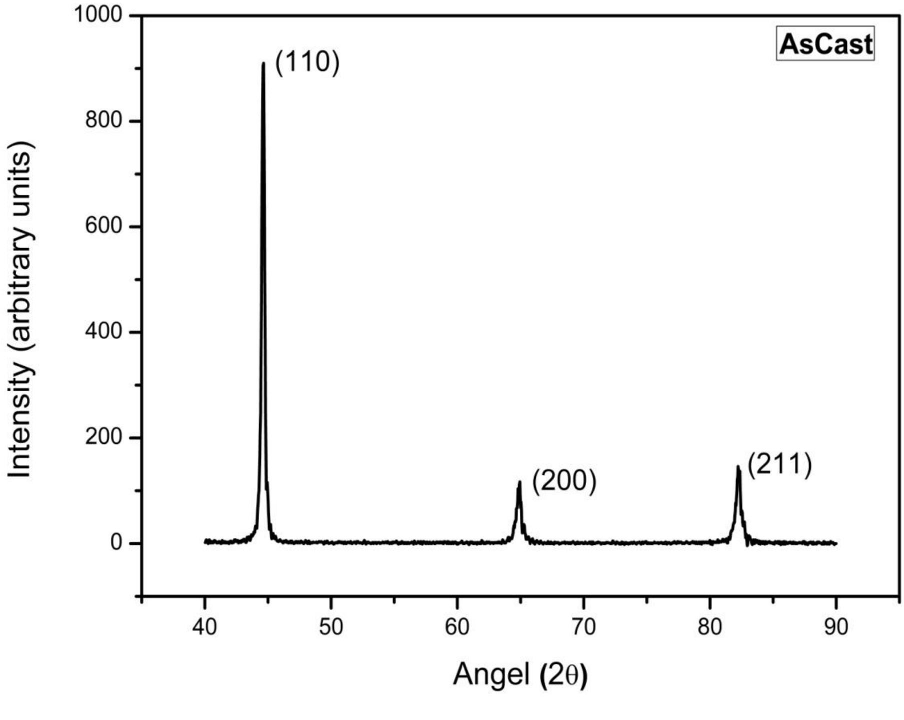
FIGURE 4.1: Microstructures of (1) As-Cast (2) Hardened &Tempered (3) Normalized (4) Annealed and (5) Austempered specimen at 200X

Table 4.1 Nodularity (%), Nodule Count and Graphite Area Fraction (%)

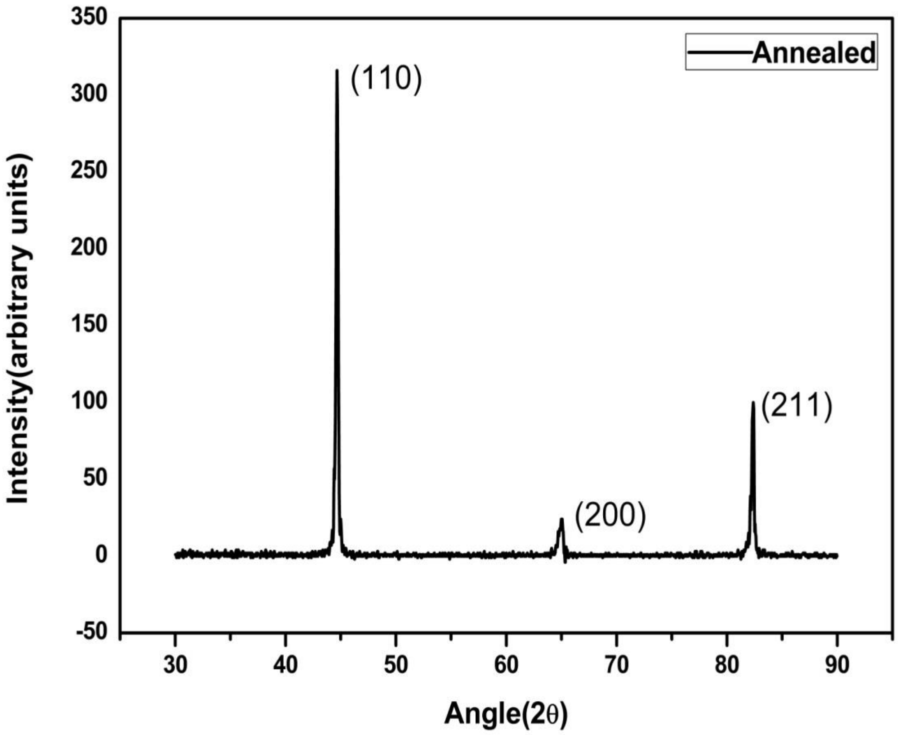
Specimen	Nodularity (%)	Nod. count	Graphite Area Fraction (%)
As-cast	93.625	1217	28.910
Annealed	98.092	1943	25.168
Normalized	98.092	1763	32.072
Hardened &Tempered	98.722	991	23.819
Austempered	96.714	2542	21.499

4.2. XRD Analysis

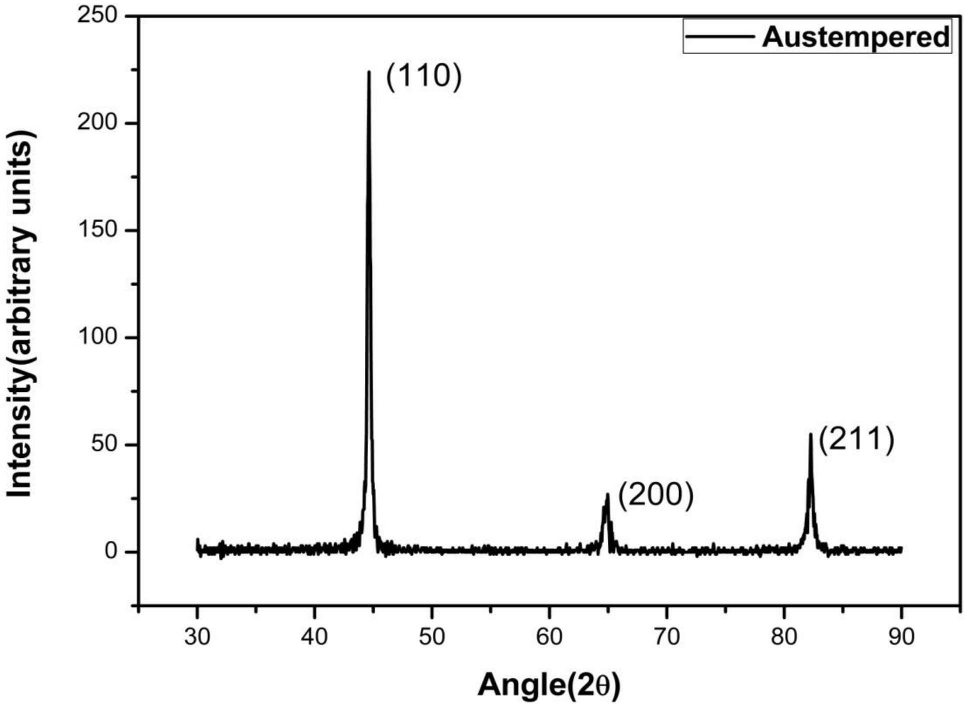
XRD of as-cast and heat treated materials were carried out (step size 2°/min. and (30-90) 2ϴ range) with X'pert PanAnlytical, model- PPW3040/00, X-ray diffractometre. Planes, Crystal structure were determined with the help of Expert High Score, JCPDS and plots were drawn by Origin Pro8.0 software. Planes were determined by matching d-spacing, 2ϴ and intensity of peaks (three) with the help of database of expert high score software. This is done for all the as-cast and the heat treated specimens. There major peaks are at angles 44.6^0, 64.8^0 &82.3^0 were obtained for as-cast and each of the heat treated specimens with a minor difference in 2ϴ. Upon analysis, BCC crystal structure was obtained for as-cast as well as each heat treated specimens. However, the intensity of respective peak was found to be changing. The least intensity was obtained for austempered specimen and that of as-cast specimen was highest for the planes (110), (200) & (211) at respective angles.



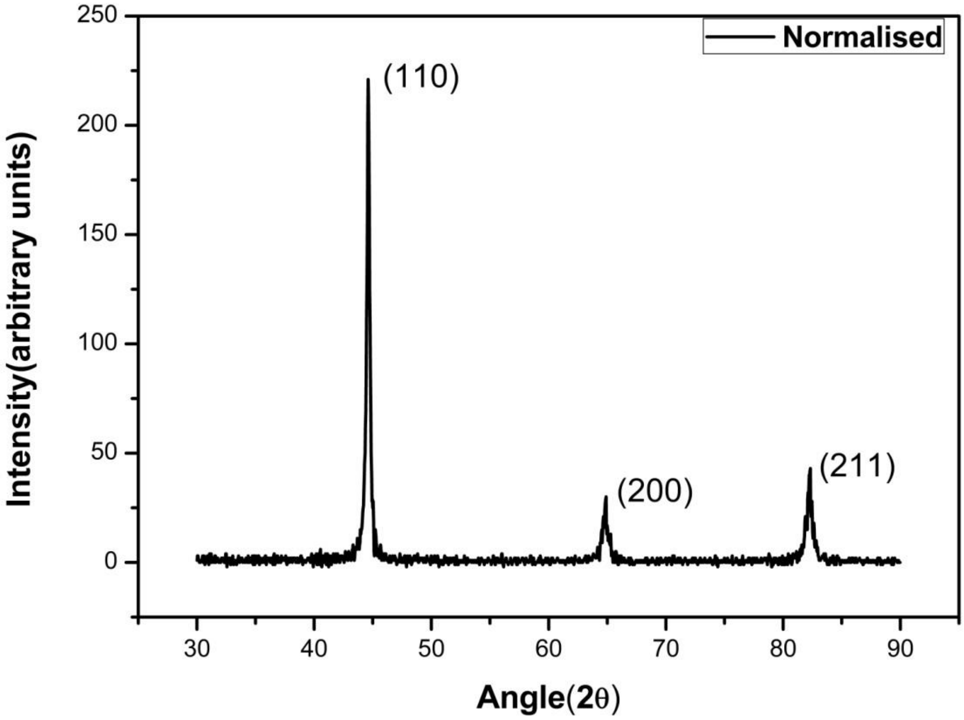
(a)


Annealed
(110)
(200)
(211)
Intensity(arbitrary units)
Angle(2θ)

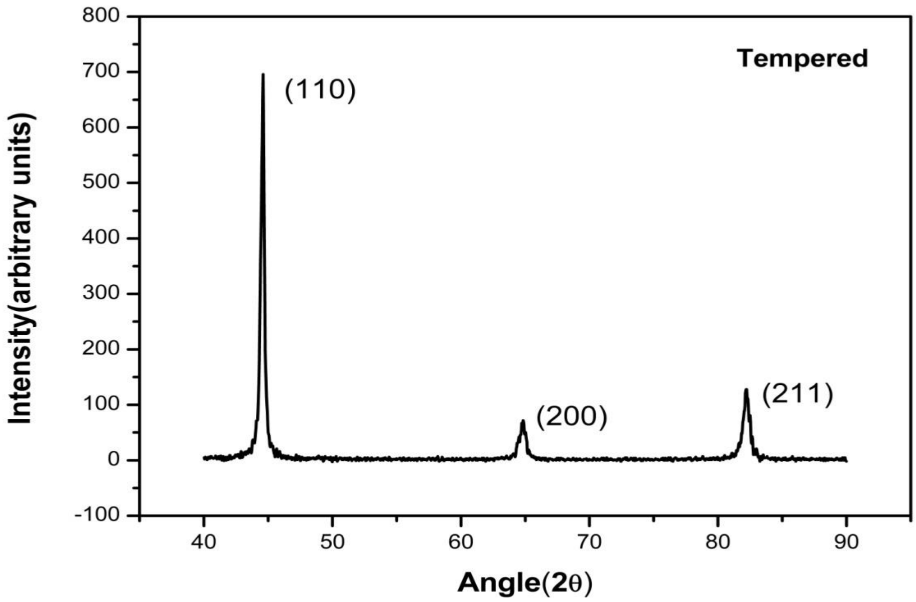
(b)


Austempered
(110)
(200)
(211)
Intensity(arbitrary units)
Angle(2θ)

(c)



(d)



(e)

Fig.4.2:XRD images of (a) As-cast (b) Annealing (c) Austempering (d) Normalizing (e) Hardening & Tempering specimens

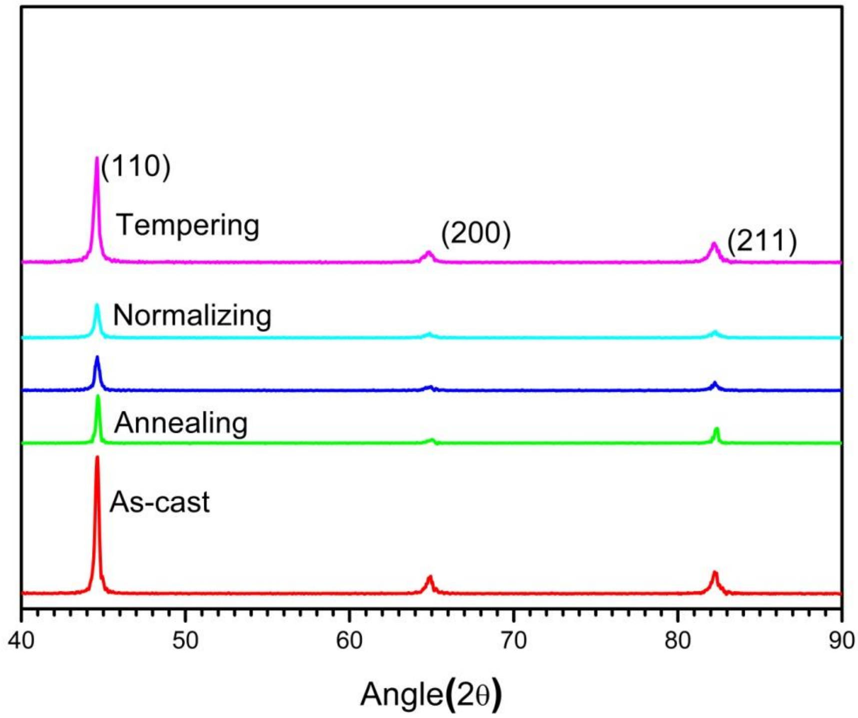


Fig.4.3: Comparison of XRD images of as-cast, annealed, austempered, normalized and hardened & tempered specimens

TABLE 4.2 Planes, Crystal size, Crystal Structure and Residual Stain

Specimen	Planes	Crystal size(nm)	Crystal Structure	Residual strain(%)
Tempering	(110),(200),(211)	225	BCC	0.342
Annealing	(110),(200),(211)	123	BCC	0.164
Austempering	(110),(200),(211)	97	BCC	0.323
As-cast	(110),(200),(211)	42	BCC	0.205
Normalizing	(110),(200),(211)	31	BCC	0.249

4.3. Mechanical Properties

The mechanical properties of as-cast and heat-treated specimen are given in Table 4.3. It was found that the hardened & tempered specimen has highest UTS of 1054 MPa with considerable amount of ductility i.e. 12.73%. The lowest ductility value of 11.9% was obtained for normalized specimen having UTS value of 691.2 MPa. The UTS value of austempered specimen was more than normalized specimen i.e. 842.5 MPa with ductility of 14.11% which is more than both normalized and hardened & tempered specimen. Results found so far was up to the excepted values of respective specimens. On the other hand the annealed and as-cast specimens showed least strength value of 236.1 MPa and 359.6 MPa, with highest percentage elongation of 27.47% and 25.15% respectively. The similar kind of trend was obtained from hardness and impact energy values. The Vickers's hardness values for hardened & tempered, normalized, austempered specimens was found to be 610 HV20, 508 HV20 & 425 HV20 with impact energy value of 9.15 J, 7.63 J & 10.15 respectively. The Vicker's hardness value for as-cast and annealed specimens was 277 HV20 and 220 HV20 respectively. However, the impact energy values for both of the specimen could not determined because of very high ductility and beyond the capacity of the Izod impact tester. The highest UTS value for hardened & tempered specimen is due to the hard tempered martensitic microstructure. The fair amount of ductility of 12.73% was achieved because of removal of residual strain during tempering treatment which was occurred during quenching in mineral oil from such as high austenizing temperature (1000°C).

Table 4.3: Mechanical properties of heat treated and as cast specimen

Sample ID	Mechanical properties				
	UTS (MPa)	0.2% YS (MPa)	% Elongation	Hardness (HV20)	Impact Energy (J)
As-cast	359.96	160.63	32.22	277	-----
Annealed	336.1	159.7	31.89	220	-----
Normalized	691.2	245.6	11.90	508	7.63
Hardened &Tempered	1054	722.9	12.73	610	9.149
Austempered	842.5	356.7	14.11	445	10.15

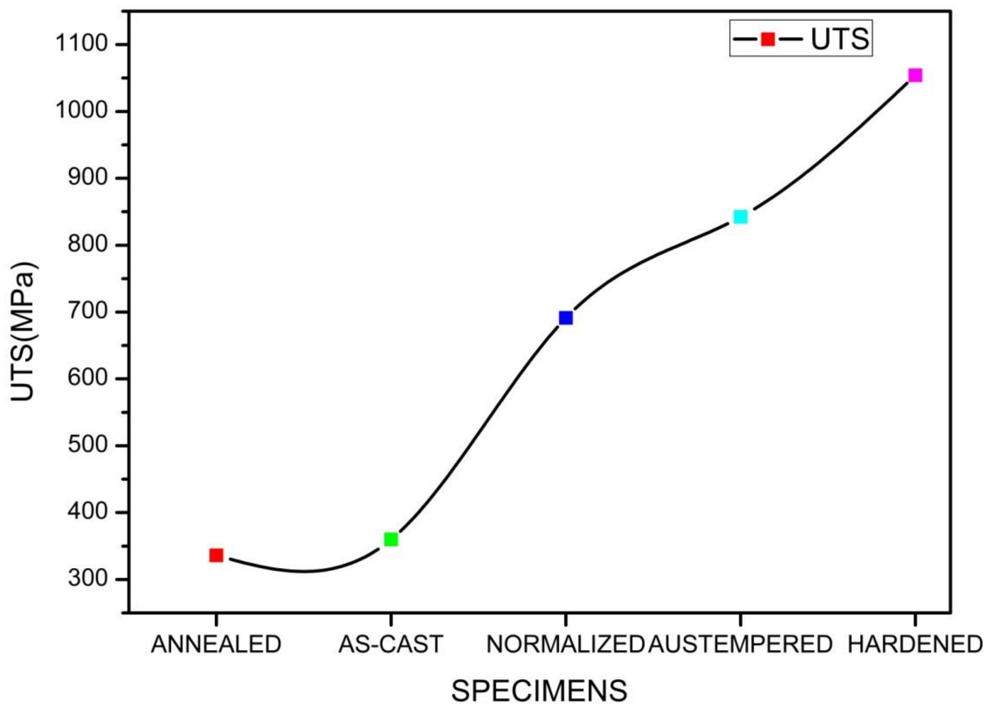


Fig.4.4: UTS of as-cast and heat treated specimen

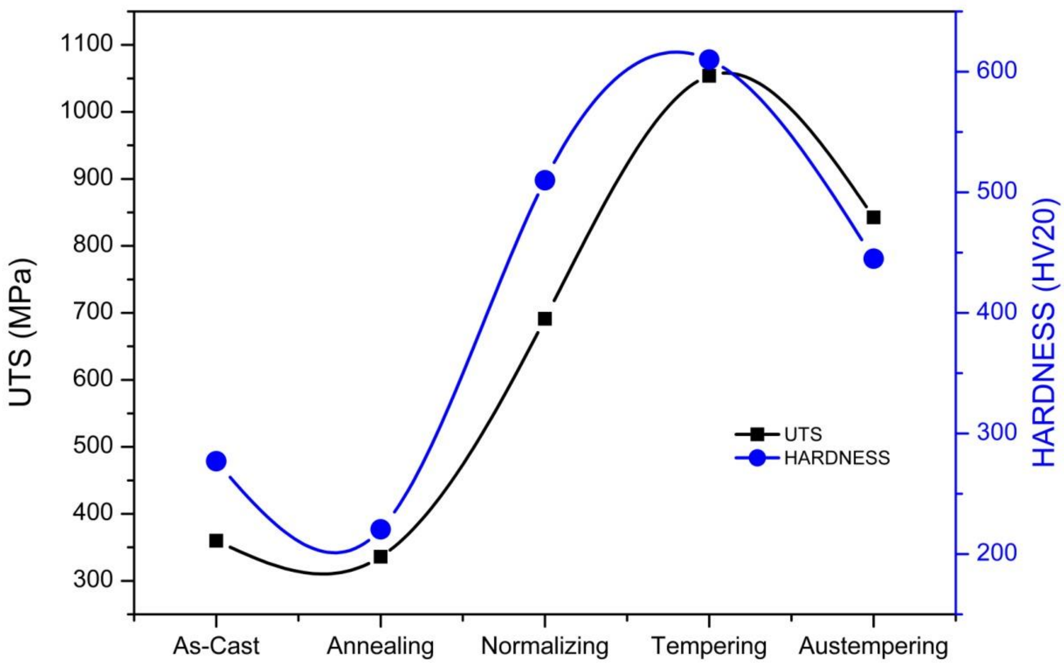


Fig.4.5: Hardness and UTS of as-cast and heat treated specimens

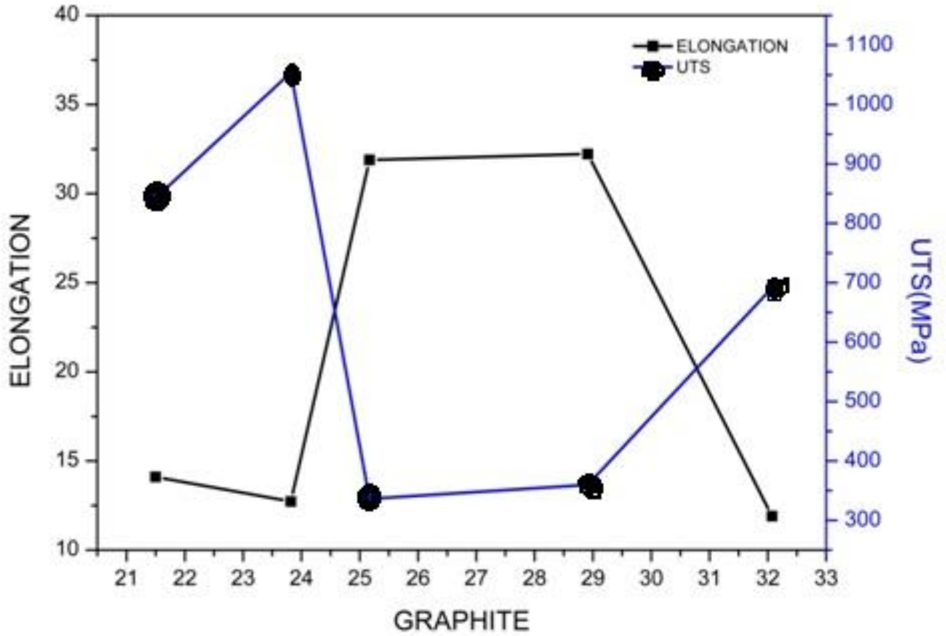


Fig. 4.6: UTS and % Elongation versus Graphite area fraction (%)

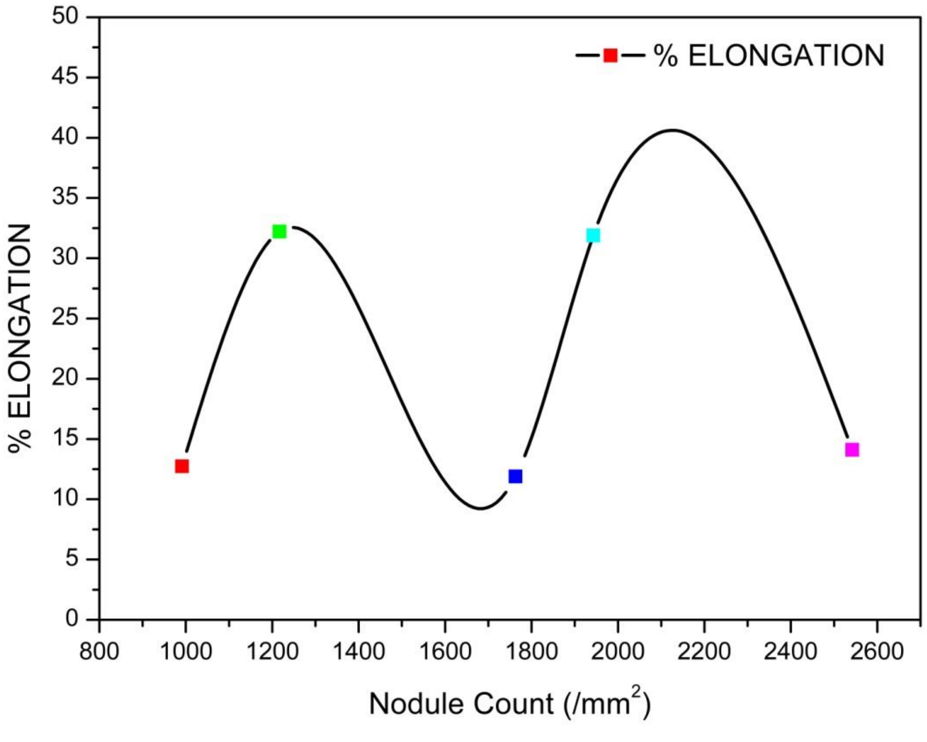


Fig.4.7: %Elongation v/s Nodule Count per mm^2

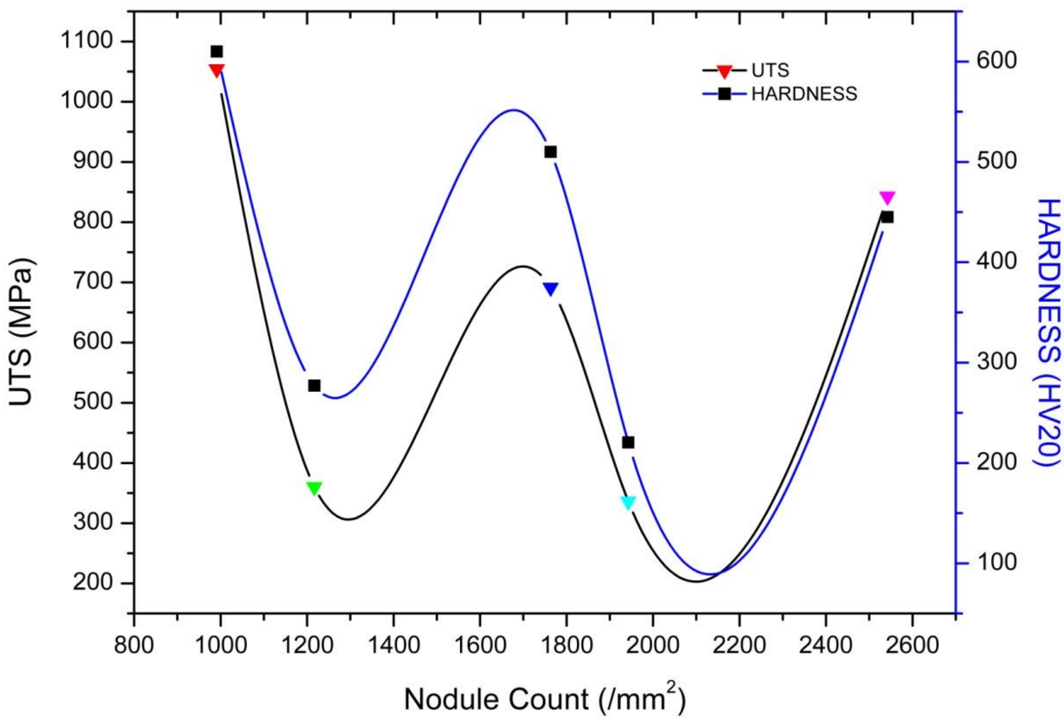
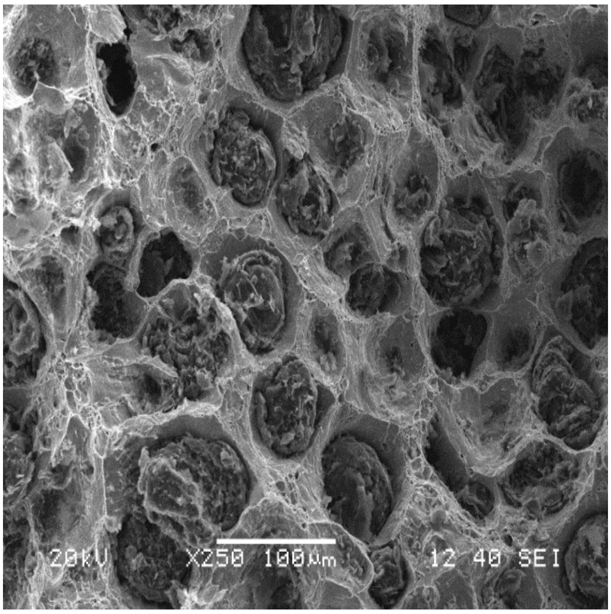


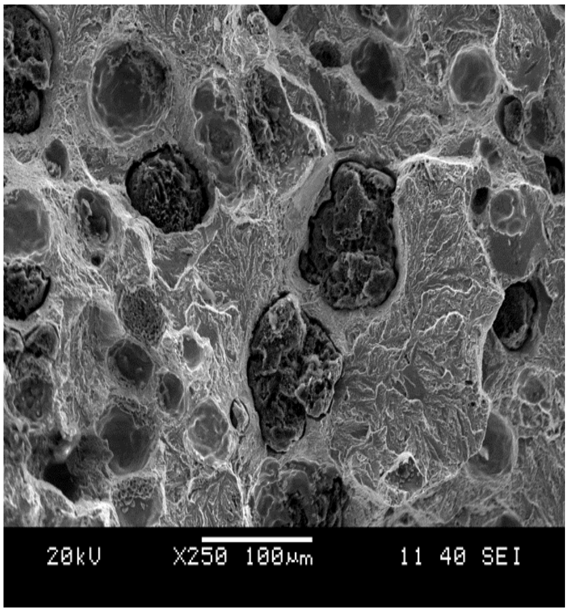
Fig.4.8: UTS and Hardness v/s Nodule Count per mm^2

4.4. Fractographic Analysis

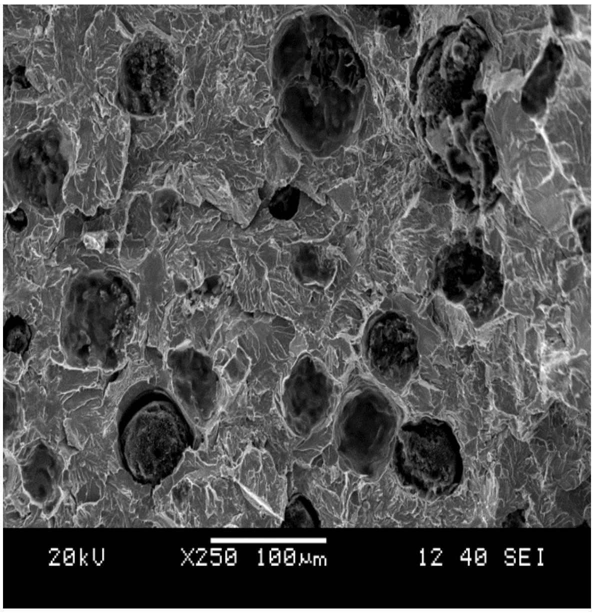
Fracture surface of heat treated and as cast specimens are observed in the Scanning Electron Microscope at 50X, 250X and 500 X magnifications and shown in figure. It is observed that annealed specimen is fully ductile. It is observed to have dimples in both the specimens as a result of formation of micro-voids and coalescence. Intergranular fracture path can be clearly observed in as-cast and annealed specimens. River markings are observed with dimples at some areas in case of normalized as well as tempered specimen. River makings are the characteristics of cleavage brittle fracture, which is low-energy fracture that propagates along well defined low-index crystallographic planes known as cleavage planes [19]. Although cleavage fracture is observed, specimens are not purely brittle rather semi-brittle with dimples at some areas.



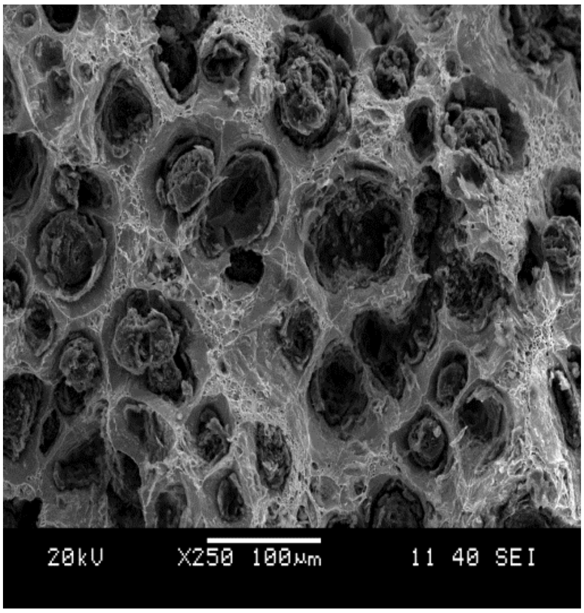
(a) As Cast



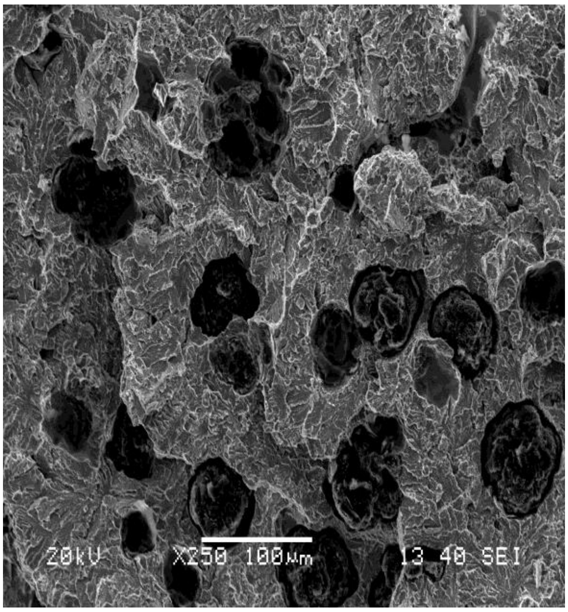
(b)Hardened & Tempered



(c)Normalized

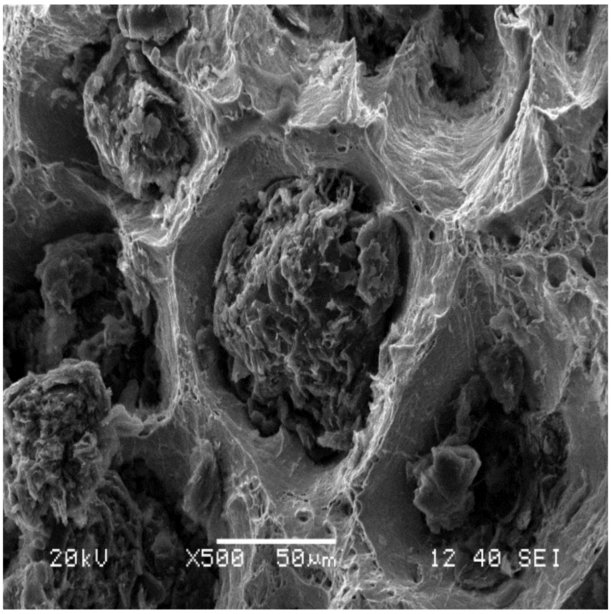


(d) Annealed

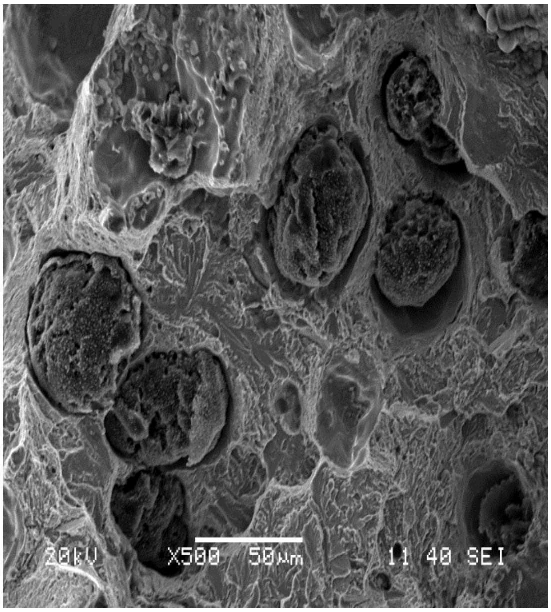


(e)Austempered

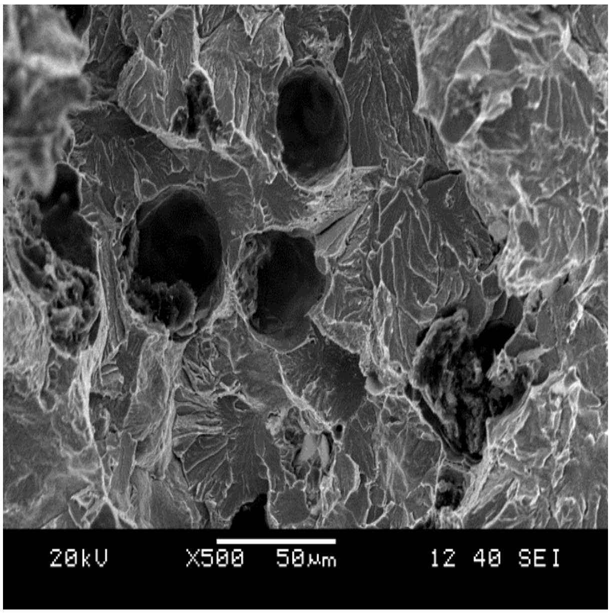
Fig.4.9: SEM images of (a) as cast (b) Hardened & Tempered (c) Normalized (d) Annealed (e) Austempered at 250x



(a) As Cast



(b) Hardened & Tempered



(c) Normalized

Fig.4.10: SEM images of (a) as cast (b) Hardened & Tempered (c) Normalized at 500X

The fracture surface of as-cast and heat treated specimen after tensile test observed under Scanning Electron Microscope (SEM) at 250X and 500X. The as-cast and annealed specimen appeared to have dimples around the nodule as well as in the matrix. The presence of dimples occurred due to the formation of micro-voids and coalescence phenomena, when specimen is under uniaxial tensile loading. The presence of dimple characterise the ductile fracture. On the other hand normalized, hardened & tempered and austempered specimens have flate, shinny fracture surface were observed with naked eye. The SEM observation reveals the presence of rivermarking in the respective matrices i.e. pearlite, tempered martensitic and ausferritic respectively characterising the brittle mode of failure [19]. However, when observed under 500X magnification shallow dimples are observed around the nodule for hardened and tempered specimens. The presence of both rivermarking as well as shallow dimples indicates towards the mix mode of fracture rather than fully brittle fracture for hardened & tempered specimen. On the other hand along with rivermarking, cleavage planes were also observed for normalized and austempered specimens [19].

Chapter 5

Conclusions

Ductile iron specimens are subjected to different heat treatment processes followed by mechanical property, microstructural and fractographic characterization study. The following conclusions are drawn from current study:

- Annealing heat treatment leads to homogeneity in the matrix, consequently improving nodularity, percentage elongation and impact energy at the expense of UTS and hardness.
- Hardened & tempered specimen has highest UTS and hardness along with fair amount of ductility.
- The UTS and hardness value for normalised and austempered specimens are in between annealed and hardened & tempered specimens with least amount of ductility for normalized specimen due to hard pearlitic matrix.
- X-ray diffraction analysis confirmed BCC crystal structure for as-cast as well as heat treated specimens.
- Along with the change in matrix heat treatment of ductile iron also enhances the quantitative metallographic aspect such as nodularity and nodule count.
- The as-cast and annealed specimen with fully ferritic matrix showed fully ductile fracture characterised by microvoid coalescence and dimple rupture phenomena.
- The hardened & tempered specimen observed to have both rivermarking and shallow dimples indicating mixed mode fracture.
- The normalized and austempered specimen have rivermarking as well as cleavage plane, charactering low energy brittle fracture.

Chapter 6

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